# STATISTICAL METHODS ATTACHMENT WILCOXON RANK-SUM TEST

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When a single compliance well group is being compared to background data and a non-parametric test is needed, the Kruskal-Wallis procedure should be replaced by the Wilcoxon Rank-Sum test (Lehmann, 1975; also known as the two-sample Mann-Whitney U test). For most ground-water applications, the Wilcoxon test should be used whenever the proportion of nondetects in the combined data set exceeds 15 percent. However, to provide valid results, do not use the Wilcoxon test unless the compliance well and background data groups both contain at least four samples each.

To run the Wilcoxon Rank-Sum Test, use the following algorithm. Combine the compliance and background data and rank the ordered values from 1 to N. Assume there are n compliance samples and m background samples so that N=m+n. Denote the ranks of the compliance samples

by  $C_i$  and the ranks of the background samples by  $B_i$ . Then add up the ranks of the compliance samples and subtract n(n+1)/2 to get the Wilcoxon statistic W:

$$W = \sum_{i=1}^{n} C_i - \frac{1}{2} n(n+1).$$

The rationale of the Wilcoxon test is that if the ranks of the compliance data are quite large relative to the background ranks, then the hypothesis that the compliance and background values came from the same population should be rejected. Large values of the statistic W give evidence of contamination at the compliance well site.

To find the critical value of W, a Normal approximation to its distribution is used. The expected value and standard deviation of W under the null hypothesis of no contamination are given by the formulas

$$E(W) = \frac{1}{2}mn;$$
  $SD(W) = \sqrt{\frac{1}{12}mn(N+1)}$ 

An approximate Z-score for the Wilcoxon Rank-Sum Test then follows as:

$$Z = \frac{W - E(W) - \frac{1}{2}}{SD(W)}.$$

The factor of 1/2 in the numerator serves as a continuity correction since the discrete distribution of the statistic W is being approximated by the continuous Normal distribution.

Once an approximate Z-score has been computed, it may be compared to the upper 0.01 percentile of the standard Normal distribution,  $z_{.01}=2.326$ , in order to determine the statistical significance of the test. If the observed Z-score is greater than 2.326, the null hypothesis may be rejected at the 1 percent significance level, suggesting that there is significant evidence of contamination at the compliance well site.

#### **EXAMPLE 13**

The table below contains copper concentration data (ppb) found in water samples at a monitoring facility. Wells 1 and 2 are background wells and well 3 is a single compliance well suspected of contamination. Calculate the Wilcoxon Rank-Sum Test on these data.

Copper Concentration (ppb)				
Backg	round	Compliance		
Well 1	Well 2	Well 3		
4.2	5.2	9.4		
5.8 11.3	6. <del>4</del> 11.2	10.9 14.5		
7.0 7.3	11.5 10.1	16.1 21.5		
8.2	9.7	17.6		
	Backg Well 1  4.2 5.8 11.3 7.0 7.3	Background Well 1 Well 2  4.2 5.2 5.8 6.4 11.3 11.2 7.0 11.5 7.3 10.1		

#### SOLUTION

Step 1. Rank the N=18 observations from 1 to 18 (smallest to largest) as in the following table.

Ranks of Copper Concentrations				
Backs	ground	Compliance		
Well 1	Well 2	Well 3		
1	2	8		
3	4	11		
13	12	15		
5	14	16		
6	10	18		
7	9	17		
	Backs Well 1	Background Well 1 Well 2  1 2 3 4 13 12 5 14 6 10		

- Step 2. Compute the Wilcoxon statistic by adding up the compliance well ranks and subtracting n(n+1)/2, so that W=85-21=64.
- Step 3. Compute the expected value and standard deviation of W.

E(W) = 
$$\frac{1}{2}$$
mn = 36  
SD(W) =  $\sqrt{\frac{1}{12}}$ mn(N+1) =  $\sqrt{114}$  = 10.677

Step 4. Form the approximate Z-score.

$$Z = \frac{W - E(W) - \frac{1}{2}}{SD(W)} = \frac{64 - 36 - 0.5}{10.677} = 2.576$$

Step 5. Compare the observed Z-score to the upper 0.01 percentile of the Normal distribution. Since Z=2.576>2.326=z.01, there is significant evidence of contamination at the compliance well at the 1 percent significance level.

### 3.2.1 Handling Ties in the Wilcoxon Test

Tied observations in the Wilcoxon test are handled in similar fashion to the Kruskal-Wallis procedure. First, midranks are computed for all tied values. Then the Wilcoxon statistic is computed as before but with a slight difference. To form the approximate Z-score, an adjustment is made to the formula for the standard deviation of W in order to account for the groups of tied values. The necessary formula (Lehmann, 1975) is:

$$SD^*(W) = \sqrt{\frac{mn(N+1)}{12} \left(1 - \sum_{i=1}^{g} \frac{t_i^3 - t_i}{N^3 - N}\right)}$$

where, as in the Kruskal-Wallis method, g equals the number of groups of distinct tied observations and t<sub>i</sub> represents the number of tied values in the ith group.

## STATISTICAL METHODS ATTACHMENT POISSON PREDICTION LIMITS

### 2.2.4 Poisson Prediction Limits

To estimate a Prediction limit at a particular well using the Poisson model, the approach described by Gibbons (1987b) and based on the work of Cox and Hinkley (1974) can be used. In this case, an upper limit is estimated for an interval that will contain all of k future measurements of an analyte with confidence level 1- $\alpha$ , given n previous background measurements.

To do this, let  $T_n$  represent the sum of the Poisson counts of n background samples. The goal is to predict  $T_k$ , representing the total Poisson count of the next k sample measurements. As

Cox and Hinkley show, if  $T_n$  has a Poisson distribution with mean  $\mu$  and if no contamination has occurred, it is reasonable to assume that  $T_k^*$  will also have a Poisson distribution but with mean  $c\mu$ , where c depends on the number of future measurements being predicted.

In particular, Cox and Hinckley demonstrate that the quantity

$$\left[T_{k}^{*} - \frac{c(T_{n} + T_{k}^{*})}{(1+c)}\right]^{2} \frac{c(T_{n} + T_{k}^{*})}{(1+c)^{2}}$$

has an approximate standard Normal distribution. From this relation, an upper prediction limit for  $T_k^*$  is calculated by Gibbons to be approximately

$$T_{k}^{a} = cT_{k} + \frac{ct^{2}}{2} + ct\sqrt{T_{k}\left(1 + \frac{1}{c}\right) + \frac{t^{2}}{4}}$$

where  $t=t_{n-1,\alpha}$  is the upper (1- $\alpha$ ) percentile of the Student's t distribution with (n-1) degrees of freedom. The quantity c in the above formulas may be computed as k/n, where, as noted, k is the number of future samples being predicted.

#### EXAMPLE 10

Use the following benzene data from six background wells to estimate an upper 99% Poisson Prediction limit for the next four measurements from a single downgradient well.

	Benzene Concentrations (ppb)						
Month	Well 1	Well 2	Well 3	Well 4	Well 5	Well 6	
1	2	<2	2	<2	<2	<2	
$\bar{2}$	2	<2	<b>2</b>	15.0	<2	2	
3	2	<2	<2 ⋅	<2	<2	<2	
Ă	ā	12.0	2	<b>Q</b>	<2	<2	
<u> </u>	$\bar{a}$	<2	<2	<2	<2	10.0	
6	: <b>2</b>	<u>~</u>	<u>a</u>	2	<2	. <2	
_		. •				•	

#### SOLUTION

- Step 1. Pooling the background data yields n=36 samples, of which, 33 (92%) are nondetect. Because the rate of detection is so infrequent (i.e., <10%), a Poisson-based Prediction limit may be appropriate. Since four future measurements are to be predicted, k=4, and hence, c=k/n=1/9.
- Step 2. Set each nondetect to half the detection limit or 1 ppb. Then compute the Poisson count of the sum of all the background samples, in this case,  $T_n=33(1)+(12.0+15.0+10.0)=70.0$ . To calculate an upper 99% Prediction limit, the upper 99th percentile of the t-distribution with (n-1)=35 degrees of freedom must be taken from a reference table, namely  $t_{35,01}=2.4377$ .
- Step 3. Using Gibbons' formula above, calculate the upper Prediction limit as:

$$T_{k}^{*} = \frac{1}{9}(70) + \frac{(2.4377)^{2}}{2(9)} + \frac{2.4377}{9}\sqrt{70(1+9) + \frac{(2.4377)^{2}}{4}} = 15.3 \text{ ppb}$$

Step 4. To test the upper Prediction limit, the Poisson count of the <u>sum</u> of the next four downgradient wells should be calculated. If this sum is greater than 15.3 ppb, there is significant evidence of contamination at the downgradient well. If not, the well may be regarded as clean until the next testing period.

The procedure for generating Poisson prediction limits is somewhat flexible. The value k above, for instance, need not represent multiple samples from a single well. It could also denote a collection of single samples from k distinct wells, all of which are assumed to follow the same Poisson distribution in the absence of contamination. The Poisson distribution also has the desirable property that the sum of several Poisson variables also has a Poisson distribution, even if the individual components are not identically distributed. Because of this, Gibbons (1987b) has suggested that if several analytes (e.g., different VOCs) can all be modeled via the Poisson distribution, the combined sum of the Poisson counts of all the analytes will also have a Poisson distribution, meaning that a single prediction limit could be estimated for the combined group of analytes, thus reducing the necessary number of statistical tests.

A major drawback to Gibbons' proposal of establishing a combined prediction limit for several analytes is that if the limit is exceeded, it will not be clear which analyte is responsible for "triggering" the test. In part this problem explains why the ground-water monitoring regulations mandate that each analyte be tested separately. Still, if a large number of analytes must be regularly tested and the detection rate is quite low, the overall facility-wide false positive rate may be unacceptably high. To remedy this situation, it is probably wisest to do enough initial testing of background and facility leachate and waste samples to determine those specific parameters present at levels substantially greater than background. By limiting monitoring and statistical tests to a few

### Draft 1/28/93

parameters meeting the above conditions, it should be possible to contain the overall facility-wide false positive rate while satisfying the regulatory requirements and assuring reliable identification of ground-water contamination if it occurs.

Though quantitative information on a suite of VOCs may be automatically generated as a consequence of the analytical method configuration (e.g., SW-846 method 8260 can provide quantitative results for approximately 60 different compounds), it is usually unnecessary to designate all of these compounds as leak detection indicators. Such practice generally aggravates the problem of many comparisons and results in elevated false positive rates for the facility as a whole. This makes accurate statistical testing especially difficult. EPA therefore recommends that the results of leachate testing or the waste analysis plan serve as the primary basis for designating reliable leak detection indicator parameters.

TABLE 6. PERCENTILES OF STUDENT'S t-DISTRIBUTION

 $(F = 1-\alpha; n = degrees of freedom)$ 

		-			<del></del>	,		
,	.60	.75	.90	.95	.975	.99	.995	.9995
1	.325	1.000	3.078	6.314	12.706	31.821	- 63.657	635.619
2	.289	.816	1.886	2.920	4.303	6.965	9.925	21.598
3	.277	.765	1.638	2.353	3.182	4.541	5.841	12.941
4	.271	.741	1.533	2.132	2.776	3.747	4.604	8.610
5	. 267	.727	1.476	2.015	2.571	3.365	4.032	6.859
6	.265	.718	1.440	1.943	2.447	3.143	3.707	5.959
7	.283	.711	1.415	1.395	2.365	2.993	3.499	5.405
8	.262	.706	2.397	1.860	2.306	2.896	3.355	5.041
9	.261	703	1.383	1.833	2.262	2.821	3.250	4.731
10	.260	.700	1.372	1.812	2.228	2.764	3.169	4.587
11	.260	.697	1.363	1.796	2.201	2.718	3.106	4.437
12	.259	.695	1.356	1.782	2.179	2.681	3.055	4.318
13	.259	.694	1.350	1.771	2.160	2.650	3.012	4.221
14	.258	.692	1.345	1.761	2.145	2.624	2.977	4.140
15	. 258	.691	1.341	1.753	2.131	2.502	2.947	4.073
16	. 258	.690	1.337	1.746	2.120	2.583	2.921	4.015
17	.257	. 689	1.333	1.740	2.110	2.567	2.898	3.965
18	.257	.688	1.330	1.734	2.101	2.552	2.878	3.922
19	.257	.688	1.328	1.729	2.093	2.539	2.861	3.883
20	. 257	687	1.325	1.725	2.086	2.523	2.845	3.850
21	. 257	. 686	   1.323	1.721	2.080	2.518	2.831	3.819
22	.256	.686	1.321	1.717	2.074	2.508	2.819	3 792
23	.256	. 585	1.319	1.714	2.069	2.500	2.807	3.767
24	.256	.685	1.318	1.711	2.064	2.492	2.797	3.745
25	.256	. 584	1.316	1.708	2.060	2.485	2.787	3.725
28	.256	. 684	1.315	1.706	2.056	2.479	2.779	3.707
27	.258	. 654	1.314	1.703	2.052	2.473	2.771	3.690
23	.256	. 683	1.313	1.701	2.048	2.467	2.763	3.674
29	. 256	. 683	1.311	1.699	2.045	2.462	2.756	3.659
30	.256	.683	1.310	1.697	2.042	2.457	2.750	3.646
40	.255	.681	1.303	1.684	2.021	2.423	2.704	3.551
60	.254	.679	1.296	1.671	2.000	2.390	2.660	3.460
120	.254	.677	1.259	1.658	1.980	2.358	2.617	3.373
-a	. 253	. 674	1.282	1.645	1.960	2.326	2.576	3.291

SOURCE: CRC Handbook of Tables for Probability and Statistics. 1966. W. H. Beyer, Editor. Published by the Chemical Rubber Company. Cleveland, Ohio.

## STATISTICAL METHODS ATTACHMENT NON-PARAMETRIC PREDICTION INTERVALS

### 4.2.1 Non-parametric Prediction Intervals

When the parametric assumptions of a Normal-based Prediction limit cannot be justified, often due to the presence of a significant fraction of nondetects, a non-parametric Prediction interval may be considered instead. A non-parametric upper Prediction limit is typically constructed in the same way as a non-parametric upper Tolerance limit, that is, by estimating the limit to be the maximum value of the set of background samples.

The difference between non-parametric Tolerance and Prediction limits is one of interpretation and probability. Given a background measurements and a desired confidence level, a non-parametric Tolerance interval will have a certain coverage percentage. With high probability, the Tolerance interval is designed to miss only a small percentage of the samples from downgradient wells. A Prediction limit, on the other hand, involves the confidence probability that the next future sample or samples will definitely fall below the upper Prediction limit. In this sense, the Prediction limit may be thought of as a 100% coverage Tolerance limit for the next k future samples.

As Guttman (1970) has indicated, the confidence probability associated with predicting that the next single observation from a downgradient well will fall below the upper Prediction limit -- estimated as the maximum background value -- is the same as the expected coverage of a similarly constructed upper Tolerance limit, namely  $(1-\alpha)=n/(n+1)$ . Furthermore, it can be shown from Gibbons (1991b) that the probability of having k future samples all fall below the upper non-parametric Prediction limit is  $(1-\alpha)=n/(n+k)$ . Table A-7 in Appendix A lists these confidence levels for various choices of n and k. The false positive rate associated with a single Prediction limit can be computed as one minus the confidence level.

Balancing the ease with which non-parametric upper Prediction limits are constructed is the fact that, given fixed numbers of background samples and future sample values to be predicted, the maximum confidence level associated with the Prediction limit is also fixed. To increase the level of confidence, the only choices are to 1) decrease the number of future values to be predicted at any testing period, or 2) increase the number of background samples used in the test. Table A-7 can be used along these lines to plan an appropriate sampling strategy so that the false positive rate can be minimized and the confidence probability maximized to a desired level.

#### EXAMPLE 17

Use the following arsenic data from a monitoring facility to compute a non-parametric upper Prediction limit that will contain the next 2 monthly measurements from a downgradient well and determine the level of confidence associated with the Prediction limit.

		Arsenic Conce	ntrations (ppb	)
_		ackground We	lls	Compliance
Month	Well 1	Well 2	Well 3	Well 4
1	<5	7	<b>්</b>	
$\hat{\mathbf{z}}$	<5	6.5	<5	
3	8	<b>්</b>	10.5	
4	<5	6	<b>්</b>	
Ś	9	12	< 5	8
6	10	<	9	14

#### SOLUTION

- Step 1. Determine the maximum value of the background data and use this value to estimate the upper Prediction limit. In this case, the Prediction limit is set to the maximum value of the n=18 samples, or 12 ppb. As is true of non-parametric Tolerance intervals, only uncontaminated wells should be used in the construction of Prediction limits.
- Step 2. Compute the confidence level and false positive rate associated with the Prediction limit. Since two future samples are being predicted and n=18, the confidence level is found to be n/(n+k)=18/20=90%. Consequently, the Type I error or false positive rate is equal to (1-.90)=10%. If a lower false positive rate is desired, the number of background samples used in the test must be enlarged.
- Step 3. Compare each of the downgradient samples against the upper Prediction limit. Since the value of 14 ppb for month 2 exceeds the limit, conclude that there is significant evidence of contamination at the downgradient well at the 10% level of significance.

# ATTACHMENT 11C AMBIENT AIR MONITORING PLAN AND PROCEDURES

#### **ATTACHMENT 11C**

#### **Ambient Air Monitoring Program**

Part 111 of the Michigan Natural Resources and Environmental Protection Act 451 (Hazardous Waste Management) requires that air emissions from hazardous waste facilities be addressed by implementation of an ambient air monitoring program. The Michigan Department of Natural Resources (MDNR) Air Quality Division has developed and published guidelines for these monitoring programs: "Network Design Criteria for Act 64 Ambient Air Monitoring Programs" (March 1985). MDNR approval of the ambient air monitoring plan is required as part of the permitting process for the Allen Park Clay Mine Landfill (APCML).

This Air Monitoring Plan describes the ambient monitoring program to be conducted when the APCML begins operation. Ambient monitoring shall be conducted on a frequent and routine basis, with a broad range of parameters being addressed. The monitoring program is intended to provide the MDNR with the data necessary to evaluate the compliance status of the facility with respect to relevant standards and to develop a long-term record of ambient air quality near the facility. This Air Monitoring Plan has been prepared based upon a review of Act 64 and pertinent MDNR documents. This document is intended to fulfill the Act 64 ambient air monitoring permit requirement.

The monitoring program is designed to collect data suitable for review for compliance with standards.

This monitoring plan describes the strategy and procedures to be followed in conducting the ambient monitoring program. This program includes the following features to ensure high quality, representative data:

- Year-round monitoring
- Monitoring for a broad range of metals
- Sensitive sampling and analysis methods
- 24-hour sampling
- Monitoring at downwind and upwind locations
- Background monitoring

- Conformance with MDNR network design criteria
- Quality assurance and quality control procedures

The remainder of this document discusses the following features of the proposed program:

- Target compound selection
- Monitoring strategy
- Sample collection and analysis procedures
- Quality assurance
- Data reduction and reporting

#### **Target Compound Selection**

Samples shall be collected and analyzed for a defined set of target compounds. The disposal of liquids in the landfill is prohibited. Also, stored wastes will be covered on a daily basis. Nevertheless, volatile organics will be monitored as part of the proposed program. Metals, total suspended particulate matter (TSP) and PM-10 (particulates with aerodynamic diameter less than 10  $\mu$ m) will also be monitored as target parameters.

The target compound list was developed based on several information sources. The following factors were considered in selecting target compounds:

- Potential emissions from the landfill
- Toxicity

The information sources reviewed include:

- Waste Acceptance Plan for the APCML
- MDNR Air Quality Division papers
- Similar monitoring programs in Michigan which are licensed or proposed for license under Act 64

Table 11C-1 (Attachment 11C) shows the Target Compound List for this program. For the purpose of this monitoring program each target compound is considered a potential emission

from the landfill. Table 11C-2 summarizes the proposed sampling and analytical methods with the detection limit goals for the methods.

#### Monitoring Strategy

The monitoring strategy discussed in this section has been developed to ensure that the objectives of the program are met. The overall objectives of the air monitoring program are to:

- Determine background ambient air concentrations near the APCML; monitor emissions attributed to landfill operations
- Comply with MDNR Network Design Criteria for Act 64 Ambient Air Monitoring Programs

The specific monitoring components which shall help achieve these objectives are the selection of appropriate sampling locations, methods, and schedules. Each of these elements is discussed below.

MDNR requires that monitoring equipment be placed at a minimum of two locations: one background station and one station at the point of highest predicted source impact concentration. APCML proposes to utilize four existing sampling stations which are located within the APCML facility. APCML proposes to locate a colocated sampling station at the maximum impact point (M2 on Figure 11C-2). A windrose which illustrates the distribution of wind speeds and wind directions for the area is shown on Figure 11C-1 (Attachment 11C). The tentative station locations are shown on Figure 11C-2 (Attachment 11C).

Each station shall be fully equipped to collect samples for each target compound. Each sampler shall be located free from obstructions and reactive surfaces in accordance with MDNR requirements. The intake of each sampling train is to be located 3 to 5 meters above ground level.

The sampling schedule for this program follows MDNR requirements for Act 64 air monitoring programs. Sampling shall be conducted on every third day for 3 months. This 3-month period shall include the season when facility emissions are potentially the highest. If this sampling program begins prior to July, the initial sampling will be conducted on a once-per-6-day frequency. The sampling frequency shall be increased to once every 3 days for the months of July, August, and September. All measurements shall be conducted over a 24-hour duration (i.e., averaging time of 24 hours).

Following the 3-month, 3-day sampling frequency period, monitoring shall be reduced to a once-per-6-day frequency, subject to MDNR approval. The MDNR may deny the reduced sampling schedule if standard operating procedures have not been followed or if the program has failed a quality assurance audit.

Landfilling activities are likely to be intermittent. The ambient air monitoring will continue for 30 days following cessation of landfill activities. Monitoring will startup again upon the receipt of the first load of waste and continue as before.

Sampling sessions shall be scheduled to coincide with total suspended particulate (TSP) sampling conducted by the U.S.EPA. The EPA's sampling also occurs once every 6 days. Sampling conducted at a 3-day frequency shall be scheduled such that every other sampling session coincides with the EPA's sampling.

#### Sample Collection Procedures

Airborne Particulate Matter (TSP and PM-10)

Total suspended particulate matter (TSP) and suspended particulates of less than ten-micron aerodynamic diameter (PM-10) shall be collected using the General Metal Works (GMW) Model 2000 H (or equivalent) high-volume sampler (hi-vol), with glass fiber filters. The TSP sampling method is an EPA reference method in general use for nearly two decades. The PM-10 method is a modification of the EPA TSP method that uses aerodynamic effects to collect only the desired (< 10 m) particulate size fraction, which involves the addition of a special, commercially available inlet adaptor to the standard hi-vol, and flow control to maintain a 40 cubic-feet-per-minute (CFM) flow rate. TSP and PM-10 samples shall be collected according to requirements listed in 40 CFR Part 50, Appendix A.

The TSP and PM-10 samples are analyzed gravimetrically by weighing each filter in a humidity-controlled environment prior to sampling, and then reweighing in that environment after sampling. The net weight gain is then combined with the measured sample air volume to determine the ambient air concentration.

#### Metals

Filter pads from the TSP monitors shall be analyzed for metals content utilizing EPA Reference Methods 6010.

Volatile Organic Compounds (VOCs)

Volatile organic compounds shall be collected on Tenax solid sorbent cartridges, SUMMA passivated canisters, or equivalent utilizing a calibrated flow-controlled sampling pump. Solid sorbent cartridges are designed to efficiently collect VOCs of varying molecular weight. A known volume of air shall be drawn through the clean cartridge at a predetermined flow rate where the compounds of interest shall be collected on the sorbent material. APCML proposes to conduct further analysis using the Gas Chromatography/Mass Spectrometry (GC/MS) or Gas Chromatography/Flame Ionization Detection (GS/FID with the Hall Detector) as an acceptable alternate. The GS/FID method shall, if employed, be conducted in accordance with EPA reference methods.

The design of this monitoring program assumes that all samples collected shall be analyzed. However, it is the nature of field programs that events occur which occasionally degrade the quality or integrity of individual samples. Therefore, only samples that satisfy specific validation criteria shall be selected for laboratory analysis. Samples that fail to meet the established criteria outlined in Table 11C-1 shall not be prioritized for analysis. Sampler operation, sample integrity, sample documentation, and meteorological conditions shall be assessed as summarized in Table 11C-3.

#### Analytical Protocols

TSP and PM-10: Gravimetric Analysis

The preweighed filters shall be returned to the air quality laboratory of choice for gravimetric analysis. Particulate weights shall be measured to the nearest tenth of a milligram in accordance with standard U.S.EPA procedures.

#### Selected Metals

Five metals (As, Cd, Cr, Hg, Pb) shall be measured utilizing EPA reference method 601OA (Inductively Coupled Argon Plasma - Atomic Emission Spectroscopy).

Volatile Organic Compounds: Gas Chromatography/Mass Spectrometry (GC/MS) Analysis

Volatile organic compounds collected in solid sorbent cartridges shall be thermally desorbed and analyzed by GC/MS based on EPA Methods T-01 and T-02 from the Compendium of

Methods for the Determination of Toxic Organic Compounds in Ambient Air EPA-600/4-89/017 (revision of the original compendium EPA/600/4-84/041 and first supplement EPA/600/4-87/006). The methods involve flash vaporization of the solid sorbent cartridge employing a

heated chamber in conjunction with an inert gas purge. The inert gas transfers the thermally desorbed compounds from the cartridge in the gas phase onto a cold trap and subsequently onto a GC column held at ambient temperature. The GC column temperature is then "ramped" and the components eluting from the column are identified and quantified by mass spectrometry.

An alternative to GC/MS is Gas Chromatography with Flame Ionization Detection (GS/FID with the Hall Detector). Ambient air analysis will be conducted with acceptable methods for the SUMMA passivated canisters.

Upon receipt at the laboratory, samples from the field shall be transferred to the sample processing area. The custody seals shall be inspected prior to opening each sample shipment. Upon opening, the accompanying chain-of-custody documentation shall be verified, signed, and dated. Sample numbers shall be assigned to serve as unique sample identifiers during all subsequent sample handling and analysis. Samples shall be returned to their respective containers, sealed, and stored to await analysis.

Relatively low detection limits are required for this program for several reasons. First, many of the target compounds typically are present in ambient air. Thus, sensitive detection limits are necessary to distinguish facility contributions, if any, from background. Second, the MDNR has issued acceptable ambient concentration guidelines for many compounds. For VOCs the concentrations are relatively low, 0.02-1.0  $\mu$ g/m³. For metals, the detection limits are expected to be in the range 0.03 to 11 ng/m³ depending on the specific metal.

#### Sample Control and Chain-of-Custody

The purpose of sample control and chain-of-custody procedures is to document the identity of the sample and its handling from the point of collection through completion of sample analysis and data reduction. Custody records trace a sample from its collection through all transfers of custody until it is transferred to the analytical laboratory. Internal laboratory records then document the custody of the sample through its final disposition.

Sample control shall begin in the analytical laboratory, with the preparation of collection media. Sampling kits shall be provided to the field coordinator by the laboratory. The sampling kits shall be enclosed in coolers, and shall include the appropriate sample media, Chain-of-Custody Forms, and all appropriate shipping blanks. The sample media provided in

the sampling kits shall be packaged by the laboratory. Completed sampling kits shall be returned to the laboratory by the field coordinator after the samples have been collected. As samples are collected, each sample shall be labeled with the following information:

- Sample identification number
- Sampling site
- Date
- Time
- Sampler signature of person collecting the sample
- Remarks any pertinent field observations or further sample description

After collection, identification, and preservation, the sample shall be maintained under Chainof-Custody procedures.

In addition to the labeling of the sample media, a field sample log shall be maintained by the field coordinator (or designee) in which a complete account is kept of samples collected at each sampling site. The sample log is filled out as soon as possible after sample collection. Information entered in the log for each sample includes the following:

- Sample site location
- Sample ID number
- Sample type
- Date collected
- Start and end time of sample run

Chain-of-Custody procedures serve at least two essential purposes in ambient monitoring programs as follows:

- They provide a formalized mechanism for the assignment of responsibility for sample integrity.
- They provide objective, physical evidence of the possession history and integrity of each sample, from collection, through analysis to data reporting, which supports the validity of site investigation data.

A sample is under a person's custody if:

It is in that person's possession

- It is in that person's view, after being in his or her possession
- It was in that person's possession and he or she locked it up to prevent tampering; or
- It is in a secure area, under the control of that person

#### The following custody procedures are followed in the field:

- 1. Prior to commencement of sampling, the field coordinator shall instruct the sampling team in the Chain-of-Custody procedures.
- 2. The field sampler shall be personally responsible for the care and custody of the samples collected until they are transferred or dispatched properly.
- The field coordinator shall determine whether proper custody procedures were followed during the fieldwork and shall decide if additional samples are required.
- 4. As soon as each sample has been collected, containerized, and labeled, it is entered on the Chain-of-Custody Form. One Chain-of-Custody Form may be used for as many as 8 samples but all samples sharing a single Chain-of-Custody Form must be packaged and shipped together. The sampler must accurately and legibly complete all of the heading information on the form. For each sample, the following information shall be entered:
  - Sample identification number (must be identical to the identification number on the sample label)
  - Date and time of sample collection
  - Type of sample media
  - Analyses to be performed

#### The following procedures are followed in shipping samples and transferring custody:

- Package samples properly for shipment and dispatch to the analytical laboratory for analysis, with a separate custody record accompanying each package.
- Seal shipping containers with Chain-of-Custody tape for shipment to the laboratory. The Chain-of-Custody seal is signed and dated by the person applying the tape. The number on the Chain-of-Custody tape is recorded in the field notebook or on the sample log.
- 3. Enter the method of shipment, the courier's name(s), and other pertinent information in the "Remarks" box. A copy of the shipper's waybill or airbill is retained by the last custodian prior to shipment.

#### Sample Collection Procedures

Filters shall be transported in a labeled envelope and directly installed into the sampling unit. Prior to sampling, the high-volume filter shall be visually inspected for defects, such as pinholes, tears, creases, or lumps. Any loose particulate shall be removed with a brush. Each filter shall be numbered for identification in one of the four corners. After sampling (approximately 1,800 M³ of air), each filter shall be folded in half so that the side with deposits only touches itself. The filter shall then be inserted back into the same labeled envelope. Until the analysis is performed, filters shall remain in their sealed plastic storage bags.

#### **Analytical Procedures**

For the PM-10 and TSP samplers, analytical procedures prescribed in 40 CFR Part 50 Appendix B shall be utilized. For metals, EPA Reference Method 601OA shall be utilized. For VOCs, EPA Reference Methods TO-1 and TO-2, or Reference Method TO-3 shall be utilized.

#### Data Reduction, Validation, and Reporting

Quality control measures shall be used to ensure the generation of reliable data from sampling analysis activities. All information shall be collected and organized clearly and concisely, and reported accurately.

#### Field Data Reduction

The data collected in the field shall be recorded on the appropriate field data sheets and/or in the field logbook, and shall be reviewed by at least two field sampling team members. Errors or discrepancies shall be noted in the field logbook. All data and calculations shall be checked by the project field coordinator or designee and shall be signed to demonstrate that the data have been reviewed and approved. All checks and data reviews are completed before data shall be given to the laboratory for calculations.

#### Laboratory Analysis Data Reduction

Analysis results shall be reduced to the appropriate concentration units. All calculations shall be recorded in the laboratory notebook, and checked and signed by the project laboratory coordinator or designee to demonstrate that the calculations

and data have been approved and reviewed. All appropriate blank corrections shall be applied to data before they are released from the laboratory.

#### **Data Validation**

Data validation is the process of filtering data and accepting or rejecting it on the basis of sound criteria. Validation methods and criteria appropriate to the type of data and the purpose of the measurement shall be used in this program. Records of all data shall be maintained, even those judged to be "outlying" or spurious values. The persons validating the data shall have sufficient knowledge of the technical work to identify questionable values.

The following criteria shall be used to evaluate the field sampling data:

- Approved test procedure
- Properly operating and calibrated equipment
- Materials that have passed QC checks

The criteria listed below shall be used to evaluate the analytical data:

- Approved analytical procedure
- Properly operating and calibrated instrumentation

#### Identification and Treatment of Outliers

Any data point that deviates markedly from others in its set of measurements shall be investigated, however, the suspected outlier shall be recorded and retained in the data set while it is investigated. Outliers shall be identified by following the procedure for identification and treatment of outliers found in <a href="The Quality Assurance Handbook for Air Pollution Measurement Systems">The Quality Assurance Handbook for Air Pollution Measurement Systems</a>, Volume 1: A Field Guide to Environmental Quality Assurance, (EPA-600/R-94/038a).

Since an outlier may result from unique circumstances at the time of sample analysis or data collection, those persons involved in the analysis and data collection shall be consulted. They may provide an experimental reason for the outlier. Further statistical analyses shall be performed with and without the outlier to determine its effect on the conclusions.

In summary, every effort shall be made to include the outlying value in the reported data. If the value is rejected, it shall be identified as an outlier, reported with its data set, and its omission shall be noted.

#### **Data Reporting**

Following all Data Reduction and Validation Procedures, results shall be reported on all parameters measured.

#### Internal Quality Control Checks and Frequency

Quality Control checks shall be performed to ensure the collection of representative samples and the generation of valid analytical results. Table 11C-4 summarizes the data quality goals. The checks shall be performed by field and laboratory personnel throughout the program, Table 11C-5 outlines the type, number, and frequency of Quality Assurance samples for this program.

#### Sampling QC Checks

The sampling Quality Control aspects for this program shall include the following:

- Daily calibration and calibration checks for the sampling pumps for flow rate
- Colocated sampling on station M2

#### System Audits

A system audit shall be conducted at least once during the program to ensure that the elements outlined in the Project QA Plan are functioning.

#### Preventative Maintenance Procedures and Schedules

An orderly program of positive actions to prevent the failure of equipment or instruments used in the sampling and analysis segments of this program shall be followed. Sampling pump maintenance shall also follow the preventive maintenance procedures recommended by the respective manufacturers.

#### **Precision**

Precision shall be determined by the collection and analysis of replicate samples. The analysis of the replicate samples by collecting colocated TSP and VOC filter samplers

shall provide an estimate of overall measurement precision. The analysis of laboratory duplicates (replicate aliquots from one collected sample) shall enable the estimation of analytical precision. One sampling location, station M2, every 6th day shall operate TSP and VOC colocated filters. This site M2 is selected from the downwind locations in the maximum impact point of the APCML.

Precision shall be determined by the collection and analysis of colocated samples and shall be expressed as the relative percent difference (RPD), which is determined according to the following equation:

$$RPD = \frac{(X_1 - X_2)}{X} \times 100$$

where  $X_1$  and  $X_2$  are the measurement results of colocated samples. X is the average of  $X_1$  and  $X_2$ .

#### Accuracy

Accuracy shall be estimated from the analysis of "blind" QC samples where true values are known to the Laboratory QC Coordinator. Accuracy shall be expressed as percent recovery or as relative error. The formulas to calculate these values are as follows:

Percent Recovery = Measured Value x 100
True Value

Relative Error = <u>Measured Value - True Value</u> x 100 True Value

#### Completeness

Completeness shall be reported as the percentage of all measurements made whose results are judged to be valid. The procedures to be used for validating data and determining of outliers were described earlier in this QA plan. The following formula is used to estimate completeness:

$$C = \frac{V}{T} \times 100$$

Where C = percent completeness,

V = number of measurements judged valid

T = total number of measurements

#### Corrective Action

The acceptance limits for the sampling and analyses to be conducted in this program shall be those stated in the method. The corrective actions are likely to be immediate in nature, and most often shall be implemented by the laboratory or field coordinator. The corrective action usually involves recalculation, reanalysis, or repeating a sample collection run. The program corrective action policy is described in the following subsections.

#### **Immediate Corrective Action**

Specific operating procedures and checklists shall be designed to help analysts detect the need for corrective action. Often the person's experience is more valuable in alerting the operator to suspicious data or malfunctioning equipment.

If a corrective action can be taken at this point, as part of normal operating procedures, the collection of poor quality data can be avoided. Instrument and equipment malfunctions are amenable to this type of action and procedures include troubleshooting guides and corrective action suggestions. The actions taken shall be noted in field or laboratory notebooks but no other formal documentation shall be required, unless further corrective action is necessary. These on-the-spot corrective actions shall be an every day part of the QA/QC system.

Corrective action during the field sampling portion of a program is most often a result of equipment failure or an operator oversight and may require repeating a run. When equipment is discovered to be defective (i.e., pre- and post-sampling calibration check) it shall be repaired or replaced and a correction factor shall be established. If a correction factor is unacceptable, the run is repeated, Operator oversight is best avoided by having field crew members audit each other's work before and after a test. Every effort shall be made by the field coordinator to ensure that all procedures are followed.

Corrective action for analytical work shall include recalibration of instruments, reanalysis of known QC samples and, if necessary, of actual field samples. If the problem is not solved in this way, more formalized long-term corrective action may be necessary.

#### **Data Reduction and Reporting**

The MDNR "Network Design Criteria for Act 64 Ambient Air Monitoring Programs" requires that the data collected in the program be reported in a particular format (i.e. Aerometric Information Retrieval System or AIRS) and schedule. These requirements ensure that the data shall be submitted regularly on a routine schedule.

#### Monitoring Data

All PM<sub>10</sub> and metals monitoring data collected in this program shall be reported to the appropriate regulatory agency once per month. TSP data will not be reported, as there is no applicable ambient air quality standard. All data for a given month shall be submitted to MDNR, or its designated authority, in the EPA AIRS format within 30 days following the end of that month.

## TABLE 11C-1 TARGET COMPOUND LIST FOR ACT 64 AMBIENT AIR MONITORING PROGRAM

CHEMICAL				
METALS	Arsenic			
	Cadmium			
	Chromium			
	Lead			
	Mercury			
VOLATILES	Benzene			
	Carbon Tetrachloride			
	Chloroform			
	Methylene Chloride			
	Tetrachloroethene			
•	1,1,1,2-Tetrachloroethane			
	1,1,2,2-Tetrachloroethane			
	Trichloroethene			
ŕ	1,1,2-Trichloroethane			
	Toluene			
	Xylene			
PARTICULATE MATTER	Total Suspended Particulate (TSP)			
	Inhalable Particulates (PM-10)			

TABLE 11C-2 SAMPLING AND ANALYTICAL METHODS WITH DETECTION LIMIT GOALS

Sampling Method	Analytical Method	Sample Duration	Approximate Flowrate	Sample Volume	Analytical Detection Limit	Method Detection Limit
High Volume sampler	Gravimetric	24 hr.	45 cfm	1,800 m <sup>3</sup>	0.1 mg/sample	60 ng/m³
High Volume sampler with size selective inlet	Gravimetric	24 hr.	45 cfm	1,800 m <sup>3</sup>	0.1 mg/sample	60 ng/m³
Glass-fiber filter, high volume sampler	ICAP or AAS	24 hr.	45 cfm	1,800 m <sup>3</sup>	0.05-20 μg/sample	0.03-11 ng/m³
Solid sorbent cartridges or passivated canisters	GC/MS or GC/FID	24 hr.	20-100 sccm	30-150 liters	25 ng/sampie	0.02-1.0 μg/m³ (a)
cubic feet per minute n standard cubic centimet cubic meters milligrams micrograms nanograms	ers per minute		ne Carbon Tetrachle Chloroform Methylene Chlori Tetrachloroethen 1,1,1,2-Tetrachlo 1,1,2,2-Tetrachlo Trichloroethene	oride ide ne roethane roethane	rent VOCs (μg/r 0.05 0.02 0.04 0.5 0.5 0.1 0.1 0.1 0.03	π³):
	High Volume sampler  High Volume sampler with size selective inlet  Glass-fiber filter, high volume sampler  Solid sorbent cartridges or passivated canisters  cubic feet per minute standard cubic centimet cubic meters milligrams micrograms	High Volume sampler  High Volume sampler with size selective inlet  Glass-fiber filter, high volume sampler Solid sorbent cartridges or passivated canisters  GC/FID  cubic feet per minute standard cubic centimeters per minute cubic meters milligrams micrograms	High Volume sampler  High Volume sampler  with size selective inlet  Glass-fiber filter, high volume sampler  Solid sorbent cartridges or passivated canisters  GC/FID  Cubic feet per minute standard cubic centimeters per minute cubic meters milligrams micrograms  Method  Duration  Duration  Cap hr.  Cap or AAS  CAP o	High Volume sampler Gravimetric High Volume sampler with size selective inlet  Glass-fiber filter, high volume sampler Volume sampler Solid sorbent cartridges or passivated canisters  GC/FID  Cubic feet per minute standard cubic centimeters per minute cubic meters milligrams micrograms micrograms nanograms  Method Duration Flowrate  A 5 cfm  V 6 cfm  AAS  Solid sorbent cartridges or passivated canisters  Carbon Servence  Carbon Tetrachle Chloroform Methylene Chlor Tetrachloroethere 1,1,1,2-Tetrachlor Trichloroethene 1,1,2-Trichloroethene 1,1,2-Trichloroethene 1,1,2-Trichloroethene 1,1,2-Trichloroethene 1,1,2-Trichloroethene 1,1,2-Trichloroethene 1,1,2-Trichloroethene 1,1,2-Trichloroethene	High Volume sampler Gravimetric Gravimation Gravimetric Gravimation Gravimetric Gravimation Gravimetric Gravimetri	High Volume sampler         Gravimetric         24 hr.         45 cfm         1,800 m³         0.1 mg/sample           High Volume sampler with size selective inlet         Gravimetric         24 hr.         45 cfm         1,800 m³         0.1 mg/sample           Glass-fiber filter, high volume sampler         ICAP or AAS         24 hr.         45 cfm         1,800 m³         0.05-20 μg/sample           Solid sorbent cartridges or passivated canisters         GC/MS or GC/FID         24 hr.         20-100 sccm         30-150 liters         25 ng/sample           Cubic feet per minute standard cubic centimeters per minute cubic meters milligrams micrograms nanograms         (a) Minimum Detection Limits for Different VOCs (μg/mathray) for Different VOCs (μg/math

### DETROIT 1988-1992 January 1-December 31; Midnight-11 PM 10% 8% E W S WIND SPEED (KNOTS) CALM WINDS 4.10% 17-21 **NOTE:** Frequencies 1-3 indicate direction from which the wind is blowing. WINDROSE FOR DETROIT RCRA/ACT 64 OPERATING LICENSE **APPLICATION** FORD MOTOR COMPANY ALLEN PARK, MICHIGAN DWN. BY: TBM APPROVED BY: DATE: NOVEMBER 1993 PROJ. 28041.05 FILE #

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EPA ID∯ MID980568711

#### TABLE 11C-3

### EVALUATION CRITERIA FOR THE SELECTION/PRIORITIZATION OF FIELD SAMPLES FOR LABORATORY ANALYSES (PRIORITY GIVEN TO SAMPLES SATISFYING THE FOLLOWING CRITERIA)

Sampler Operation	Pre and post flow calibration     checks are in good agreemer    (<-10% difference)			
	-	on in sampler ring sampling period		
	Pre and post leak-checks are acceptable			
Sample Physical Integrity	cracks, loose	signs of damage (e.g., e caps, or other defects artridges) in the field		
	cracks, loose	signs of damage (e.g., e caps, or other defects artridges) upon receipt tory		
Sample Documentation	l) No ambiguiti identification,	es in sample , chain-of-custody, etc.		
	2) Sample I.D. t are in good a	ags and other records		

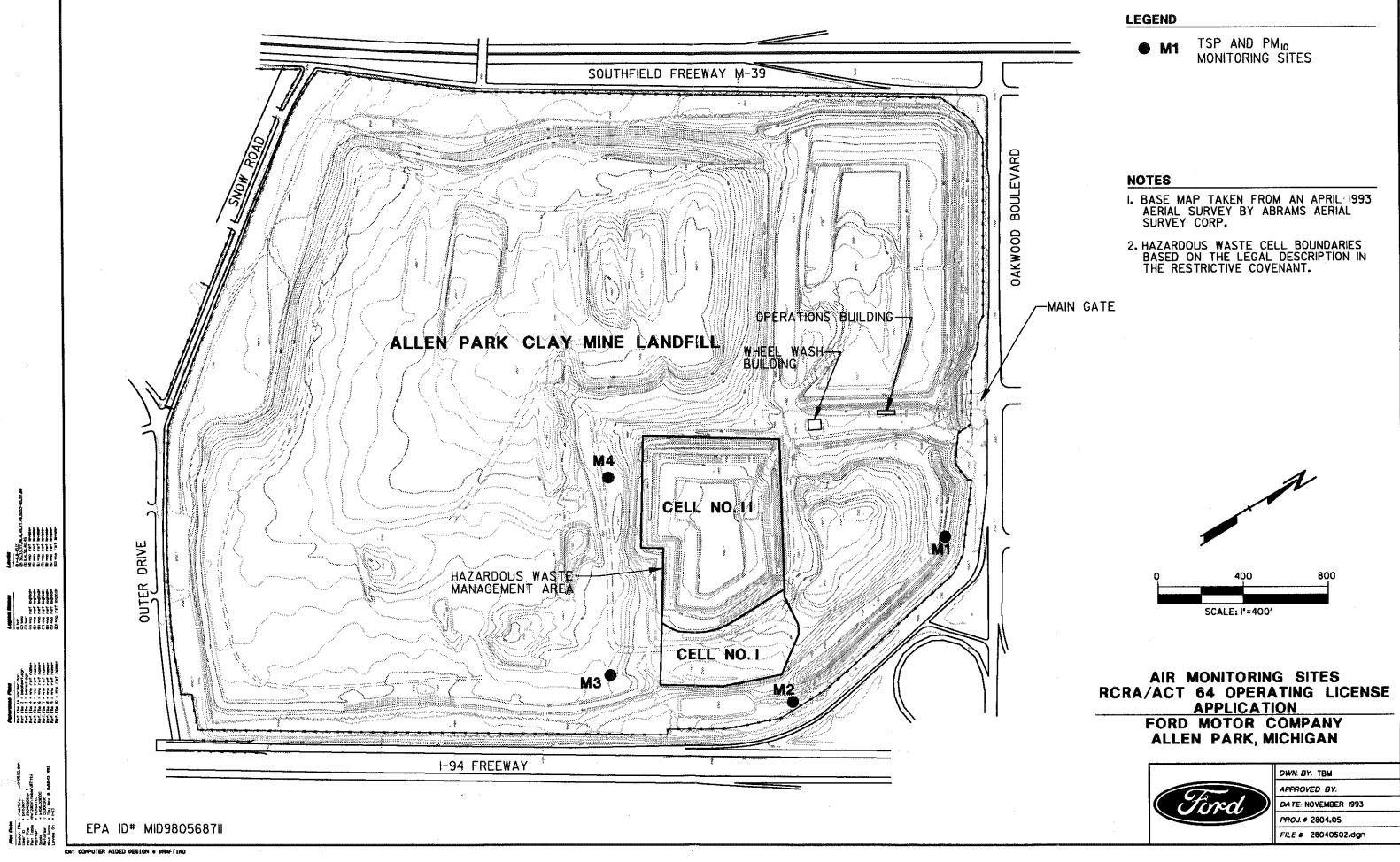
# TABLE 11C-4 SUMMARY OF DATA QUALITY GOALS

		The second secon	The second secon			
A	Objectives for Combined sampling and Analysis Procedures					
Analytical Method	Collection Matrix	Collection Matrix Precision (Std. Dev.)		Completeness		
GC/MS or GC/FID with Hall Detector	Sorbent Cartridge or Passivated Canister	± 50%	± 50%	85%		
Gravimetric	Filter	± 15%	± 15%	85%		
ICAP or AAS	Filter	± 25%	± 25%	85%		

Measurement	Sampling Objectives				
Method	Calibration Matrix	Accuracy	Completeness		
Air Flow (Sampling Pump)	Bubble Tube	± 5%	85%		
Air Flow (Hi-Vol Sampler)	Calibrated Orifice	± 7%	85%		
Air Flow (Area Sampling Pump)	Mass Flow Meter	± 5%	85%		

TABLE 11C-5
FIELD MEASUREMENT QUALITY ASSURANCE SAMPLES

Target Compound Group	Collection Matrix	Analysis Method	Collocated Samples	Samples
VOCs	Solid Sorbent Cartridges or SUMMA Passivated Canisters	GC/MS or GC/FID with Hall detector	one/day	one/day
Metals	Glass-Fiber Filter	ICAP or AAS	one/day	one/day
TSP	Glass-Fiber Filter	Gravimetric	one/day	one/day
PM-10	Glass-Fiber Filter	Gravimetric	one/day	one/day



# ATTACHMENT 11D SOIL MONITORING PLAN AND PROCEDURES

#### **ATTACHMENT 11D**

# Soil Monitoring Plan and Procedures

Soil samples will be collected on an annual basis after the initiation of filling in Cell II. Soil sample analytical parameter lists and sample collection procedures are discussed below. Proposed additional background soil sampling, to be carried out on a quarterly basis for a period of 1 year, is also discussed below. Statistical procedures for evaluating data are presented in Subsection 11D.3. A detailed laboratory QA/QC program is provided in Attachment 11A.

#### 11D.1 Soll Sampling Parameters and Schedule

# Additional Background Soil Sampling

The parameters for the proposed additional background sampling include all of the leachate monitoring parameters for which soil background values have not already been established. Table 11D-1 identifies those parameters for which background soil sampling is completed, and those parameters for which additional background sampling are proposed. The additional background sampling will consist of quarterly sampling for a period of 1 year. Each quarterly sampling event will consist of collecting one individual sample at each of the six background soil sampling locations identified on Figure 11D-2. Sample volumes, preservation methods, detection limits, holding times, and analytical methods are presented in Table 11D-3. Background soil samples will be identified by the station i.d.s used on Figure 11D-2 (DR-1-BG through DR-6-BG). Field and sample handling procedures are discussed below.

#### Operational Soil Sampling Plan

Operational soil sampling will consist of collecting six individual soil samples on an annual basis from areas off the shoulders of the facility entrance road, between the Truck Wheel Wash exit and Oakwood Boulevard. The general sampling area is shown on Figure 11D-1, and sampling locations and identification codes are presented on Figure 11D-2. In order to maintain consistency with the background soil sampling data, the sample identification codes DR-1-OP through DR-6-OP will be used to identify the operational soil sample locations, and will correspond to the foreground sampling locations identified on Figure 11D-2. During operational monitoring, samples will be collected from along the two ditches on either side of the paved entrance road, within 3 feet of the respective ditch that is closest to the paved entrance road.

The parameter list for operational soil sampling is presented in Table 11D-2. Sample preservation methods, detection limits, holding times, and analytical methods are presented in Table 11D-3. The operational monitoring parameter list (Table 11D-2) will be reviewed annually and revised if appropriate, as described in Subsection 11.3.2 of this application and in Attachment 11E, the Leachate Sampling and Monitoring Plan.

#### 11D.2 Soil Sample Collection

The procedures for soil sample collection will be as follows:

- Sample collection bottles will be provided pre-preserved from the analytical laboratory. Bottles will be prepared by labeling according to EPA Chain-of-Custody (COC) requirements using a sample tag as shown on Figure 11D-3, or equivalent. The tag will be filled out in ink and in legible handwriting. To maintain consistency with the historical record of background samples, sample identification codes DR-1-OP through DR-6-OP, and DR-1-BG through DR-6-BG, corresponding with operational and background samples, respectively, will be used.
- 2. A new piece of plastic sheeting will be spread on the ground next to each sample location in order to prevent sampling equipment from contacting the ground. A new pair of disposable PVC or latex gloves will be used by the sampler at each sampling location in order to prevent contamination of the samples. At each selected point, a stainless-steel core sampler will be used to collect a soil sample from a depth of 0 to 3 inches.
- 3. The sample bottles for volatile organic compounds will be filled first. These sample containers must be filled so that no headspace remains in the sample jar.
- The remaining sample containers will be filled in the order listed in Table 11D All sample containers will be sealed tightly, and immediately placed on ice.
- 5. All soil samples will be stored in ice coolers from the time of collection through delivery to the analytical laboratory.
- The core sampler will be decontaminated prior to sampling at each location by washing in phosphate-free soapy water and then double rinsing with distilled water.
- Field data sheets will be used to record the sampling location, time, date, and name of sampler. Copies of the field data sheets will be retained at the facility.
- 8. The Chain-of-Custody (COC) Record describing each sample will be drawn up by the sampler. This form will accompany all samples, and will be signed and dated by the sampler at the time the samples are released. The individual accepting the samples will also sign and date the same COC form acknowledging receipt of the samples.

### 11D.3 Statistical Evaluation of Soli Data

The results of the annual sampling to be conducted at the six locations along the entrance road will be compared against the background data set for the following parameters: chrome, copper, arsenic, selenium, and volatile and semi-volatile organic compounds. This list may be revised on an annual basis as described in Attachment B. The following procedures will be used to evaluate each parameter listed in the statistical evaluation program:

- Tabulate, evaluate, and reduce the existing background data. Review data to determine completeness and to determine if sample locations were consistent and clearly defined. Review any additional background data in the same manner when background data collection is completed.
- 2. Revise, if necessary, the reportable detection limits (RDLs) for each constituent. RDLs reported by analytical laboratories may change with time. Use the largest detection limit for each parameter wherever a correction for censored data is needed. Using the largest reported RDL value for each constituent is appropriate because the maximum value sets the level of accuracy that can be attained in future monitoring, even if RDL values decrease in the future.
- 3. If the background data set is 100 percent censored, then do not perform statistics. The RDL will serve as the trigger value for resampling. In this case, an operational monitoring sample result that exceeds the analytical detection limit will be confirmed by collecting an individual sample at that location and analyzing for the parameter that exceeded background. This measure is being taken in order to rule out laboratory error as a source of the detection. If the analyte is not detected in the confirmatory sample, then no further action will be taken. If the analyte is detected, then resample at that location in quadruplicate. Collect the quadruplicate samples within approximately 3 feet of each other so that the spatial variability of concentrations is tested for. Follow the procedures listed in Subsection 11.3.3 if any of the four concentrations exceed the detection level.
- 4. Assess the underlying statistical distribution of the data, and correct for log normality if necessary. After the first round of statistical evaluation has been completed, this step will consist of transforming the current data, if necessary, based on the previous evaluation. As recommended in the February 1993 USEPA Guidance document, assess normality by constructing probability plots.

Follow the attached guidance for constructing probability plots. The plotted points will approximate a straight line if the data are normal. Construct probability plots of the log-transformed and the raw data for each parameter. Compare the plots, and decide which representation of the data is closer to the normal distribution. If the log-transformed data are selected as appropriate, then transform all background and operational data for that sample point for that parameter prior to conducting any statistical tests on the data. State whether the statistical test was conducted on raw or transformed data in all reports regarding the data.

- Inspect the data set for outliers. Conduct formal testing for outliers only if a reported concentration is orders of magnitude higher than the rest of the data set. Follow the procedure for outlier testing presented at the end of this attachment. The outlier test assumes that the data other than the outlier follows a normal distribution. Therefore, if the data set is log-normally distributed (see number 3 above), then conduct the outlier test on the log-transformed data. Correct or remove an outlier from the data set only if the value can be identified as
  - a) an error in transcription or dilution;
  - b) a documented error in an analytical procedure or report of matrix interferences in the procedure; or
  - c) some other factor from those listed in the RCRA guidance (USEPA, 1989; USEPA, 1993).

In the event an outlier can be verified, obtain the MDNR's permission before removing the outlier from the data set. If no obvious cause can be identified for a value being an outlier, then it will remain in the operational data set used for statistical evaluation unless the MDNR's approval is obtained to remove it.

- 6. Inspect the current round of data for nondetects. If a parameter was reported to be below the RDL for that round, then do not perform a statistical test with that data (i.e., do not perform a statistical evaluation to determine if a nondetect represents an exceedance of background). Add the analytical result to the database for that sample point. This approach is being taken because it is reasonable to assume that a nondetect cannot represent an exceedance of background.
- 7. Evaluate the degree of censorship in the data, and select the appropriate statistical test based on this evaluation. After the first round of statistical evaluation has been completed, this step will consist of evaluating whether the statistical test used during previous rounds remains an appropriate choice for the data set. The following steps will be followed in determining how censored data will be handled and in choosing the statistical test to be performed for each sampling point:
  - a) If the percentage of nondetects in the database for the sample point is less than 15 percent, then substitute a value of 1/2 the RDL for all nondetects and calculate a prediction interval according to the procedure included at the end of this Attachment.
  - b) If the percentage of nondetects in the database for the sample point is between 15 and 50 percent, use Cohen's or Aichison's adjustment to calculate the mean and standard deviation of the background data. Use these adjusted statistics to calculate a prediction interval. Follow the procedures presented at the end of this attachment to determine which of Cohen's or Aichison's adjustment should be used. Calculate the prediction interval according to the presented procedure.

- c) If the percentage of nondetects in the database for the sample point is between 50 and 90 percent, use the Wilcoxan Rank-Sum Test to compare operational monitoring results to background data. Procedures are attached.
- d) If the percentage of nondetects in the database for the sample point is 90 percent or greater, calculate a Poisson prediction limit. Procedures are included at the end of this Attachment.
- e) If the percentage of nondetects in the background data set is 100 percent follow the procedure listed above in step number 3.
- 8. If the statistical test performed for step number 7 above indicates that an exceedance has occurred, follow the steps outlined in Subsection 11.3.3 of the Permit.

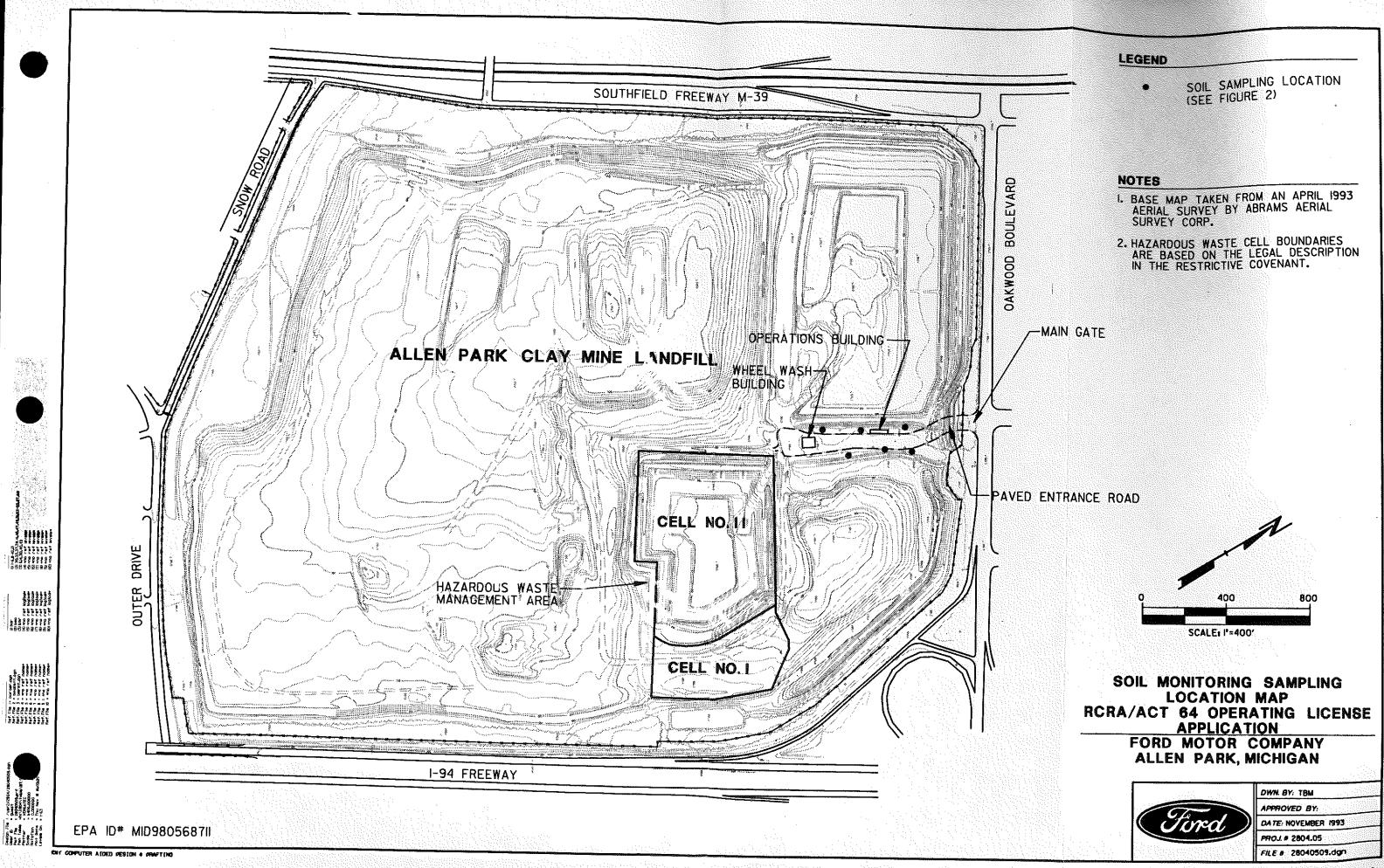


	TABLE 11D-1	
BACI	KGROUND SOIL SAMPLING PARA	METERS
Parameters	for which Soil Background Sampling	is Completed:
Arsenic Barium Cadmium Chromium Copper	Cyanide Lead Mercury Naphthalene Nickel	Phenol Selenium Silver Zinc
Proposed	Additional-Background-Soil-Samplin	g Parameters:
Antimony Beryllium Calcium Chloride Cobalt Iron Magnesium	pH Sodium Sulfate Thallium Tin Total recoverable phenolics Vanadium	
Scan :	7 Polynuclear Aromatic Hydrocarbon	s (PAHs):
Acenaphthene Acenaphthylene Anthracene Benzo(A)anthracene Benzo(A)pyrene Benzo(B)fluoroanthene	Benzo(G,H,I)perylene Benzo(K)fluoranthene Chrysene Dibenzo(A,H)anthracene Fluoranthene Fluorene	Indeno(1,2,3-CD)pyrene Naphthalene Phenanthrene Pyrene
	Volatile Organic Compounds (VOC	s)
Benzene Bromodichloromethane Bromoform Bromomethane Carbon tetrachloride Chlorobenzene Chloroethane 2-Chloroethylvinyl ether Chloroform Chloromethane Dibromochloromethane	1,2-Dichlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene Dichlorodifluoromethane 1,1-Dichloroethane 1,2-Dichloroethene trans-1,2-Dichloroethene 1,2-Dichloropropane cis-1,3-Dichloropropene trans-1,3-Dichloropropene	Ethylbenzene Methylene chloride 1,1,2,2-Tetrachloroethane Tetrachloroethene Toluene 1,1,1-Trichloroethane 1,1,2-Trichloroethane Trichloroethene Trichlorofluoromethane Vinyl chloride

# NOTES:

 VOCs include purgeable halocarbons and aromatics listed in Appendix A to Part 136 (40 CFR) - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater.

	TABLE 11D-2				
SOIL OPERATIONAL MONITORING PARAMETERS					
	Total Metals				
Antimony Arsenic Barium Beryllium Cadmium Chromium	Cobalt Copper Lead Mercury Nickel Selenium	Silver Thallium Tin Vanadium Zinc			
	Inorganics and Other Paramete	rs			
Calcium Chloride Cyanide	Iron Magnesium pH	Sodium Sulfate Total recoverable phenolics			
S	can 7 Polynuclear Aromatic Hydrod	:arbons			
Acenaphthene Acenaphthylene Anthracene Benzo(A)anthracene Benzo(A)pyrene Benzo(B)fluoroanthene	Benzo(G,H,I)perylene Benzo(K)fluoranthene Chrysene Dibenzo(A,H)anthracene Fluoranthene Fluorene	Indeno(1,2,3-CD)pyrene Naphthalene Phenanthrene Pyrene			
	Volatile Organic Compounds (VO	Cs)			
Benzene Bromodichloromethane Bromoform Bromomethane Carbon tetrachloride Chlorobenzene Chloroethane 2-Chloroethylvinyl ether Chloroform Chloromethane Dibromochloromethane	1,2-Dichlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene Dichlorodifluoromethane 1,1-Dichloroethane 1,2-Dichloroethane 1,1-Dichloroethene trans-1,2-Dichloroethene 1,2-Dichloropropane cis-1,3-Dichloropropene trans-1,3-Dichloropropene	Ethylbenzene Methylene chloride 1,1,2,2-Tetrachloroethane Tetrachloroethene Toluene 1,1,1-Trichloroethane 1,1,2-Trichloroethane Trichloroethene Trichloroethene Trichlorofluoromethane Vinyl chloride			

# NOTES:

- VOCs include purgeable halocarbons and aromatics listed in Appendix A to Part 136 (40 CFR) Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater. This list will be reviewed annually and revised if appropriate as discussed in 1.
- 2. Subsection 11.3.2.

TABLE 11D-3
SOIL SAMPLING PARAMETERS AND METHODS

Parameter	Analytical Method <sup>1</sup>	Reportable Detection Limit	Container & Preservative	Holding Time
Antimony	6010	2.5 mg/kg	G	6 months
Arsenic	7061	0.5 mg/kg	G	6 months
Barlum	6010	1 mg/kg	G	6 months
Beryllium	-6020	0.2 mg/kg	· `G	6 months
Cadmium	6010	0.5 mg/kg	G	6 months
Chromium	6010	2.0 mg/kg	G	6 months
Cobalt	6010	5.0 mg/kg	G	6 months
Copper	6010	1 mg/kg	G	6 months
Lead	6010	5.0 mg/kg	G	6 months
Mercury	7471	0.1 mg/kg	G	38 days
Nickel	6010	5 mg/kg	G	38 days
Selenium	7741	0.5 mg/kg	G	6 months
Silver	6020	0.25 μg/kg	G	6 months
Thallium	6010	1.0 mg/kg	G	6 months
Tin	6010	5.0 mg/kg	G	6 months
Vanadium	6010	1.0 mg/kg	G	6 months
Zinc	6010	1.0 mg/kg	G	6 months
Iron	6010	2.5 mg/kg	G	6 months
Bicarbonate alkalinity	310.1*	200 mg/kg	G	None specified
Carbonate alkalinity	310.1*	200 mg/kg	G	None specified
Calcium	6010	50 mg/kg	G	6 months
Cyanide	4500	2.5 mg/kg	Р	14 days
Magnesium	6010	50 mg/kg	G	6 months
Sodium	6010	50 mg/kg	G	6 months
Chloride	325.2*	20 mg/kg	G	None specified
Sulfate	375.4*	40 mg/kg	G	None specified
Total recoverable phenolics	9065	0.2 mg/kg	G	None specified
Benzene	8260	0.005 mg/kg	G, T, R	14 days
Ethylbenzene	8260	0.005 mg/kg	G, T, R	14 days
Toluene	8260	0.005 mg/kg	G, T, R	14 days

TABLE 11D-3
SOIL SAMPLING PARAMETERS AND METHODS

	SOIL SAMPLING PARAMETERS AND METHODS				
Parameter	Analytical Method <sup>1</sup>	Reportable Detection Limit	Container & Preservative	Holding Time	
Vinyl chloride	8260	0.010 mg/kg	G, T, R	14 days	
Bromodichloromethane	8260	0.005 mg/kg	G, T, R	14 days	
Bromoform	8260	0.005 mg/kg	G, T, R	14 days	
Bromomethane	8260	0.010-mg/kg	G, T, R	¹,4 days	
Carbon tetrachloride	8260	0.005 mg/kg	G, T, R	14 days	
Chlorobenzene	8260	0.005 mg/kg	G, T, R	14 days	
Chloroethane	8260	0.010 mg/kg	G, T, R	14 days	
2-Chloroethylvinyl ether	8260	0.005 mg/kg	G, T, R	14 days	
Chloroform	8260	0.005 mg/kg	G, T, R	14 days	
Chloromethane	8260	0.010 mg/kg	G, T, R	14 days	
Dibromochloromethane	8260	0.005 mg/kg	G, T, R	14 days	
1,2-Dichlorobenzene	8260	0.005 mg/kg	G, T, R	14 days	
1,3-Dichlorobenzene	8260	0.005 mg/kg	G, T, R	14 days	
1,4-Dichlorobenzene	8260	0.005 mg/kg	G, T, R	14 days	
Dichlorodifluoromethane	8260	0.010 mg/kg	G, T, R	14 days	
1,1-Dichloroethane	8260	0.005 mg/kg	G, T, R	14 days	
1,2-Dichloroethane	8260	0.005 mg/kg	G, T, R	14 days	
1,1-Dichloroethene	8260	0.005 mg/kg	G, T, R	14 days	
trans-1,2-Dichloroethene	8260	0.005 mg/kg	G, T, R	14 days	
1,2-Dichloropropane	8260	0.005 mg/kg	G, T, R	14 days	
cis-1,3-Dichloropropene	8260	0.005 mg/kg	G, T, R	14 days	
trans-1,3-Dichloropropene	8260	0.005 mg/kg	G, T, R	14 days	
Methylene chloride	8260	0.010 mg/kg	G, T, R	14 days	
1,1,2,2-Tetrachloroethene	8260	0.005 mg/kg	G, T, R	14 days	
Tetrachloroethene	8260	0.005 mg/kg	G, T, R	14 days	
1,1,1-Trichloroethane	8260	0.005 mg/kg	G, T, R	14 days	
1,1,2-Trichloroethane	8260	0.005 mg/kg	G, T, R	14 days	
Trichlorofluoromethene	8260	0.010 mg/kg	G, T, R	14 days	
Acenaphthene	8270	0.200 mg/kg	G, R	F	
Acenaphthylene	8270	0.200 mg/kg	G, R	F	

TABLE 11D-3
SOIL SAMPLING PARAMETERS AND METHODS

Parameter	Analytical Method <sup>1</sup>	Reportable Detection Limit	Container & Preservative	Holding Time
Vinyl chloride	8260	0.010 mg/kg	G, T, R	14 days
Bromodichloromethane	8260	0.005 mg/kg	G, T, R	14 days
Bromoform	8260	0.005 mg/kg	G, T, R	14 days
Bromomethane	-· 8260	0.010-mg/kg	G, T, R	1,4 days
Carbon tetrachloride	8260	0.005 mg/kg	G, T, R	14 days
Chlorobenzene	8260	0.005 mg/kg	G, T, R	14 days
Chioroethane	8260	0.010 mg/kg	G, T, R	14 days
2-Chloroethylvinyl ether	8260	0.005 mg/kg	G, T, P	14 days
Chloroform	8260	0.005 mg/kg	G, T, R	14 days
Chloromethane	8260	0.010 mg/kg	G, T, R	14 days
Dibromochloromethane	8260	0.005 mg/kg	G, T, A	14 days
1,2-Dichlorobenzene	8260	0.005 mg/kg	G, T, R	14 days
1,3-Dichlorobenzene	8260	0.005 mg/kg	G, T, R	14 days
1,4-Dichlorobenzene	8260	0.005 mg/kg	G, T, R	14 days
Dichlorodifluoromethane	8260	0.010 mg/kg	G, T, R	14 days
1,1-Dichloroethane	8260	0.005 mg/kg	G, T, A	14 days
1,2-Dichloroethane	8260	0.005 mg/kg	G, T, R	14 days
1,1-Dichloroethene	8260	0.005 mg/kg	G, T, R	14 days
trans-1,2-Dichloroethene	8260	0.005 mg/kg	G, T, R	14 days
1,2-Dichloropropane	8260	0.005 mg/kg	G, T, R	14 days
cis-1,3-Dichloropropene	8260	0.005 mg/kg	G, T, R	14 days
trans-1,3-Dichloropropene	8260	0.005 mg/kg	G, T, R	14 days
Methylene chloride	8260	0.010 mg/kg	G, T, R	14 days
1,1,2,2-Tetrachloroethene	8260	0.005 mg/kg	G, T, R	14 days
Tetrachloroethene	8260	0.005 mg/kg	G, T, R	14 days
1,1,1-Trichloroethane	8260	0.005 mg/kg	G, T, R	14 days
1,1,2-Trichloroethane	8260	0.005 mg/kg	G, T, R	14 days
Trichlorofluoromethene	8260	0.010 mg/kg	G, T, R	14 days
Acenaphthene	8270	0.200 mg/kg	G, R	F
Acenaphthylene	8270	0.200 mg/kg	G, R	F

TABLE 11D-3

# SOIL SAMPLING PARAMETERS AND METHODS

Parameter	Analytical Method <sup>1</sup>	Reportable Detection Limit	Container & Preservative	Holding Time
Anthracene	8270	0.250 mg/kg	G, R	F
Benzo(A) anthracene	8270	0.200 mg/kg	G, R	F
Benzo(A) pyrene	8270	0.200 mg/kg	G, R	F
Benzo(8)fluoranthene	-8270	0.200-mg/kg	-G, R	·F
Benzo(G,H,I)fluoranthene	8270	0.330 mg/kg	G, R	F
Benzo(K)fluoranthene	8270	0.250 mg/kg		
Chrysene	8270	0.200 mg/kg	G, R	F
Dibenzo(A,H) anthracene	8270	0.330 mg/kg	G, R	F
Fluoranthene	8270	0,300 mg/kg	G, R	F
Fluorene	8270	0.200 mg/kg	G, R	F
Indeno(1,2,3-CD) pyrene	8270	0.330 mg/kg	G, R	F
Naphthalene	8270	0.200 mg/kg	G, R	F
Phenanthrene	8270	0.200 mg/kg	G, R	F
Pyrene	8270	0.200 mg/kg	G, R	F

# NOTES:

- Test Methods for Evaluating Solid Waste, SW-846, Third Edition.
- 2 USEPA Methods 600/4-79/200.
- \* Requires performance of ASTM Leach (D3987-85) prior to wet chemistry analysis.
- P Plastic.
- G Glass.
- R Refrigeration.
- N Nitric acid to pH <2.
- T Teflon®-lined cap.
- E 7 days to extraction and 40 days from extraction.
- S Sulfuric acid to pH <2.
- AG Amber glass.
- C Hydrochloric acid to pH <2.





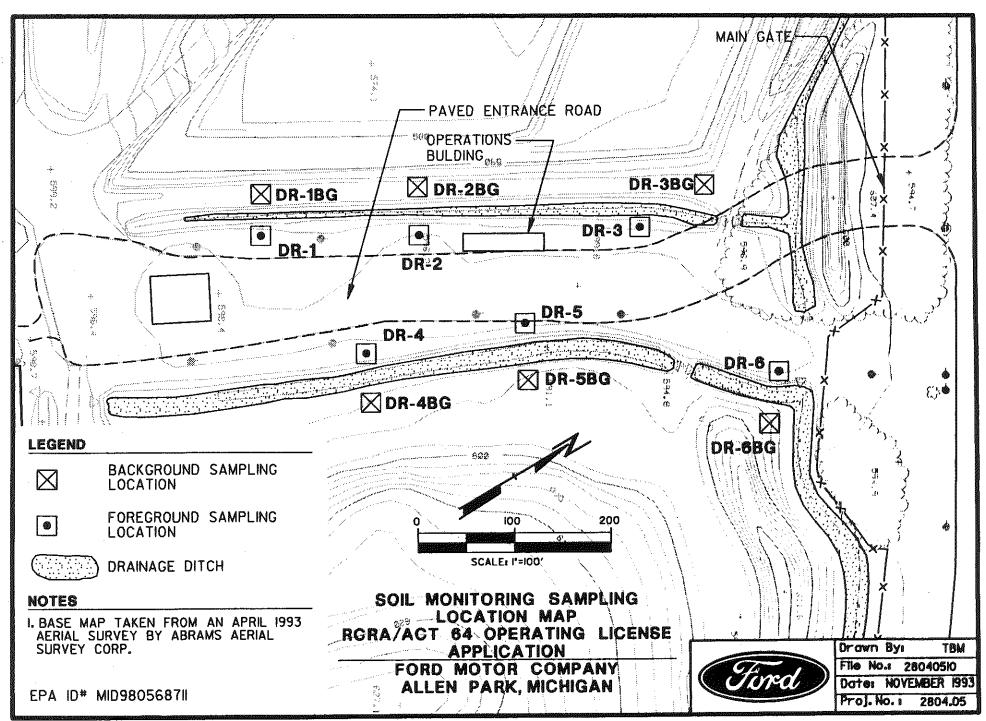


FIGURE 11D-2

		Ford Allen Park Clay Mine Ford Motor Company Entrance Road Soil Sample
	Date	•
	Time	
	Location	
((<))	Analyze for	
	Preserved with	
$\rightarrow$		•
// //	Samplers Signatures	
// //	Time	Date

EACH SAMPLE TRANSFERRED SHOULD HAVE A LABEL ATTACHED.

SAMPLE LABEL
FORD MOTOR COMPANY
ALLEN PARK, MICHIGAN



DWN. BY: TBM APPROVED BY:

DATE: NOVEMBER 1993

PROJ. # 2804.05

# ATTACHMENT 11E LEACHATE SAMPLING AND MONITORING PLAN

#### ATTACHMENT 11E

#### Leachate Sampling and Monitoring Plan

Representative leachate samples from Cells I and II will be collected on a quarterly basis. Leachate sample analytical parameter lists and sample collection procedures are discussed below. A detailed laboratory QA/QC program is provided in Attachment 11A.

#### 11E.1 Leachate Monitoring Plan

An individual leachate sample will be collected from the Cell I leachate sump on a quarterly basis and will be sampled for the list of parameters presented in Table 11E-1. This parameter list is based on the historical sampling program used at Cell I.

An individual leachate sample will be collected from the Cell II leachate sump on a quarterly basis and will be sampled for the list of parameters presented in Table 11E-2. This parameter list is based on the anticipated waste stream for Cell II. This list will be reviewed and revised annually. The review will consist of comparing waste codes for all material placed in Cell II over the previous four quarters with the list (Table 11E-2). Any of the RCRA IX parameters which appear on the waste codes and are not on the Cell II parameter list will be added to Table 11E-2. A letter report detailing the results of the review and recommendations for subsequent revisions to the operational monitoring parameter lists for soil, sediment, surface water, and the leak detection system will be submitted to the WMD for approval. This report will be submitted to the WMD within 90 days of the end of each year of operation of Cell II. The parameter lists for Cell I leachate and for the lysimeters will not undergo annual review because waste accepted into Cell II of the facility will not affect these sampling points. Leachate preservation methods, detection limits, holding times, and analytical methods are listed in Table 11E-3.

# 11E.2 Leachate Sample Collection

The procedures for leachate sample collection will be as follows:

 Sample collection bottles will be provided pre-preserved from the analytical laboratory. Prepare bottles by labeling according to EPA Chain-of-Custody (COC) requirements using a sample tag as shown on Figure 11E-1, or equivalent. The tag will be filled out in ink and in legible handwriting. To maintain consistency with the historical record of background samples, sample identification codes L1 and L2, corresponding with leachate from Cell I and Cell II, respectively, will be used.

- Spread a new piece of plastic sheeting on the ground next to each sump in order to prevent sampling equipment from contacting the ground. A new pair of disposable PVC or latex gloves will be used by the sampler at each sampling location in order to prevent contamination of the samples.
- Collect samples using disposable bailers, pre-cleaned teflon bailers, or dedicated teflon bailers:
  - a) If disposable bailers are used, a new disposable bailer will be used at each sampling point, and a new piece of nylon rope will be used to lower the bailer into the sump.
  - b) —— If pre-cleaned bailers are used, each bailer will be used at one sampling location per round. These bailers will be precleaned according to the following procedures:
    - . Brush with soapy (phosphate free soap) water and soak for a minimum of 4 hours
    - . Rinse with potable water for 3 minutes
    - . Rinse with 10 percent nitric acid solution
    - Rinse with deionized water
    - . Oven dry
      - Seal in polypropylene plastic to prevent contamination prior to use
  - c) If dedicated bailers are used, each leachate sampling point will have a bailer dedicated for use only at that sampling point. The bailer will be labeled and stored in the leachate sump while not in use by suspending the bailer from the sump cover or sidewall, so that the bailer hangs in the sump, above the leachate. Nylon rope will be used to lower the dedicated bailer in order to collect a sample. The dedicated bailer will be rinsed with leachate prior to sample collection.
- 4. Sample vials for volatile organics should be filled first. The sample should be collected in a manner which minimizes sample disturbance (i.e., if using a bailer, slowly lower the bailer into the leachate. The leachate stream should be allowed to strike the inner wall of the vial to minimize formation of air bubbles. Fill the sample vial or bottle with a minimum of splashing. Fill each vial until the water forms a positive meniscus at the brim. Allow the vial to overflow slightly, then replace the cap by gently setting it on the water meniscus. Tighten firmly, but do not over-tighten.
- 5. Invert the vial and tap lightly to check for air bubbles.
- 6. Place samples on ice immediately.

- 7. Fill appropriate bottles for other sample parameters. Note that leachate samples are not field filtered for any analytical parameters. Place samples in coolers with ice immediately following sample collection. All samples will be stored in ice coolers from time of collection through delivery to the analytical laboratory.
- 8. Fill a disposable plastic sample cup with 200 to 300 mL of leachate and collect temperature, pH and specific conductance measurements. These measurements will be collected with a Corning Model M90 pH and conductivity meter according to the manufacturer's specifications. The pH and conductivity probes will be cleaned between sampling locations by double rinsing with distilled water. The Model M90 meter will be calibrated using the two-point method once prior to use for every 4 hours of use in the field, according to the attached manufacturer's specifications for calibration. Manufacturer's specifications for instrument use and calibration are included at the end of this Attachment.
- 9. Field measurements will be recorded on field data sheets, which will identify the sampling location, time, date, and sampler, along with the measurements collected and a description of the number ant type of sample bottles filled. Copies of the field data sheets will be retained at the facility.
- 10. The Chain-of-Custody (COC) Form describing the sample will be drawn up by the sampler. This form will accompany all samples, and be signed and dated by the sampler at the time the samples are released. The individual accepting the samples will also sign and date the same COC form acknowledging receipt of the samples. Copies of the COC will be retained at the facility.

#### 11E.3 Leachate Monitoring Data Set

After each leachate monitoring sample analysis is provided by the laboratory, the results will be inspected for completeness and tabulated with the existing leachate data set.

TABLE 11E-1					
CELLI	LEACHATE MONITORING PARAN	METERS			
	Total Metals				
Arsenic Barium Cadmium Chromium	Copper Iron Lead Mercury	Nickel Selenium Silver Zinc			
	Inorganics and Other Parameters				
Bicarbonate alkalinity Carbonate alkalinity BOD COD	Calcium Chloride Cyanide Magnesium pH*	Sodium Specific conductance* Sulfate TOC Total recoverable phenolics			
	Scan 8 Phenolics				
2-Chlorophenol 4-Chloro-3-methylphenol 2,4-Dichlorophenol 2,4-Dimethylphenol 2,4-Dinitrophenol	2-Methyl-4,6-dinitrophenol 2-Nitrophenol 4-Nitrophenol	Pentachlorophenol Phenol 2,4,5-Trichlorophenol 2,4,6-Trichlorophenol			
Scan 7 Polynuclear Aromatic Hydrocarbons					
Acenaphthene Acenaphthylene Anthracene Benzo(A)anthracene Benzo(A)pyrene Benzo(B)fluoroanthene	Benzo(G,H,I)perylene Benzo(K)fluoranthene Chrysene Dibenzo(A,H)anthracene Fluoranthene Fluorene	Indeno(1,2,3-CD)pyrene Naphthalene Phenanthrene Pyrene			

# NOTES:

- Specific conductance and pH are measured in the field. Leachate samples are analyzed for total metals and are therefore not field-filtered.

	TABLE 11E-2					
CELL II LEACHATE MONITORING PARAMETERS						
	Total Metals					
Antimony Arsenic Barium Beryllium Cadmium Chromium	Cobalt Copper Lead Mercury Nickel Selenium	Silver Thallium Tin Vanadium Zinc				
	Inorganics and Other Parameters					
Bicarbonate alkalinity Carbonate alkalinity Calcium Chloride	Cyanide Iron Magnesium pH*	Sodium Specific conductance* Sulfate Total recoverable phenolics				
Sci	an 7 Polynuclear Aromatic Hydrocar	bons				
Acenaphthene Acenaphthylene Anthracene Benzo(A)anthracene Benzo(A)pyrene Benzo(B)fluoroanthene	Benzo(G,H,I)perylene Benzo(K)fluoranthene Chrysene Dibenzo(A,H)anthracene Fluoranthene Fluorene	Indeno(1,2,3-CD)pyrene Naphthalene Phenanthrene Pyrene				
	Volatile Organic Compounds (VOCs	)				
Benzene Bromodichloromethane Bromoform Bromomethane Carbon tetrachloride Chlorobenzene Chloroethane 2-Chloroethylvinyl ether Chloroform Chloromethane Dibromochloromethane	1,2-Dichlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene Dichlorodifluoromethane 1,1-Dichloroethane 1,2-Dichloroethene trans-1,2-Dichloroethene 1,2-Dichloropropane cis-1,3-Dichloropropene trans-1,3-Dichloropropene	Ethylbenzene Methylene chloride 1,1,2,2-Tetrachloroethane Tetrachloroethene Toluene 1,1,1-Trichloroethane 1,1,2-Trichloroethane Trichloroethene Trichlorofluoromethane Vinyl chloride				

# NOTES:

- Specific conductance and pH are measured in the field.
- 1.
- Leachate samples are analyzed for total metals and are therefore not field-filtered.

  VOCs include purgeable halocarbons and aromatics listed in Appendix A to Part 136

  (40 CFR) Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater. 2.
- This list will be reviewed annually and revised if appropriate as discussed in 3. Subsection 11.5.

TABLE 11E-3
LEACHATE SAMPLING PARAMETERS AND METHODS

Parameter	Analytical Method <sup>1</sup>	Estimated Detection Limit	Container & Preservative	Holding Time
Antimony (total)	6020	1 µg/L	G, N	6 months
Arsenic (total)	6020, 7061	1 μg/L	G, N	6 months
Barium (total)	6020	5 μg/L	G, N	6 months
Beryllium (total)	6020	1-µg/L	~G; N	6 months
Cadmium (total)	6020	20 μg/L	G, N	6 months
Chromium (total)	6020	20 μg/L	G, N	6 months
Cobalt (total)	6010	15 μg/L	G, N	6 months
Copper (total)	6010	20 µg/L	G, Ň	6 months
Lead (total)	6020	50 μg/L	G, N	6 months
Mercury (total)	7470	0.2 μg/L	G, N	38 days
Nickel (total)	6020	50 μg/L	G, N	6 months
Selenium (total)	6020, 7741	1 μg/L	G, N	6 months
Silver (total)	6020	0.5 μg/L	G, N	6 months
Thallium (total)	6020	2.0 μg/L	G, N	6 months
Tin (total)	200.7 <sup>2</sup>	500 μg/L	G, N	6 months
Vanadium (total)	6010	10 μg/L	G, N	6 months
Zinc (total)	6020	50 μg/L	G, N	6 months
Iron (total)	6010	0.02 mg/L	G, N	6 months
Bicarbonate alkalinity	310.1	5 mg/L	P, R	14 days
Carbonate alkalinity	310.1	10 mg/L	P, R	14 days
Calcium	6010	1 mg/L	G, N	6 months
Cyanide	335.2	5 μg/L	P, H	14 days
Magnesium	6010	1 mg/L	G, N	6 months
Sodium	6010	1 mg/L	G, N	6 months
Chloride	35.2	1 mg/L	P, R	28 days
Sulfate	375.4	2 mg/L	P, R	28 days
Total recoverable phenolics	9065	0.01 mg/L	AG, T, S	28 days
Benzene	8260	1.0 mg/L	C, G, T, R	14 days
Ethylbenzene	8260	1.0 mg/L	C, G, T, Fl	14 days
Toluene	8260	1.0 mg/L	C, G, T, R	14 days

TABLE 11E-3

LEACHATE SAMPLING PARAMETERS AND METHODS

Parameter	Analytical Method <sup>1</sup>	Estimated Detection Limit	Container & Preservative	Holding Time
Vinyl chloride	8260	5.0 mg/L	C, G, T, R	14 days
Bromodichloromethane	8260	1.0 mg/L	C, G, T, R	14 days
Bromoform	8260	1.0 mg/L	C, G, T, R	14 days
Bromomethane	8260	5.0-mg/L	C, G, T, R	∴14 days
Carbon tetrachloride	8260	1.0 mg/L	C, G, T, R	14 days
Chlorobenzene	8260	1.0 mg/L	C, G, T, R	14 days
Chloroethane	8260	5.0 mg/L	C, G, T, R	14 days
2-Chloroethylvinyl ether	8260	5.0 mg/L	C, G, T, R	14 days
Chloroform	8260	1.0 mg/L	C, G, T, R	14 days
Chloromethane	8260	5.0 mg/L	C, G, T, R	14 days
Dibromochloromethane	8260	1.0 mg/L	C, G, T, A	14 days
1,2-Dichlorobenzene	8260	1.0 mg/L	C, G, T, R	14 days
1,3-Dichlorobenzene	8260	1.0 mg/L	C, G, T, R	14 days
1,4-Dichlorobenzene	8260	1.0 mg/L	C, G, T, R	14 days
Dichlorodifluoromethane	8260	5.0 mg/L	C, G, T, R	14 days
1,1-Dichloroethane	8260	1.0 mg/L	C, G, T, R	14 days
1,2-Dichloroethane	8260	1.0 mg/L	C, G, T, R	14 days
1,1-Dichloroethene	8260	1.0 mg/L	C, G, T, R	14 days
trans-1,2-Dichloroethene	8260	1.0 mg/L	C, G, T, R	14 days
1,2-Dichloropropane	8260	1.0 mg/L	C, G, T, R	14 days
cis-1,3-Dichloropropene	8260	1.0 mg/L	C, G, T, R	14 days
trans-1,3-Dichloropropene	8260	1.0 mg/L	C, G, T, R	14 days
Methylene chloride	8260	5.0 mg/L	C, G, T, R	14 days
1,1,2,2-Tetrachloroethene	8260	1.0 mg/L	C, G, T, R	14 days
Tetrachloroethene	8260	1.0 mg/L	C, G, T, R	14 days
1,1,1-Trichloroethane	8260	1.0 mg/L	C, G, T, R	14 days
1,1,2-Trichloroethane	8260	1.0 mg/L	C, G, T, R	14 days
Trichloroethene	8260	1.0 mg/L	C, G, T, R	14 days

TABLE 11E-3

LEACHATE SAMPLING PARAMETERS AND METHODS

Parameter	Analytical Method¹	Estimated Detection Limit	Container & Preservative	Holding Time
2,4,5-Trichlorophenol	8270	10 μg/L	AG, R	E
2,6,4-Trichlorophenol	8270	10 µg/L	AG, R	E
2,4-Dichlorophenol	8270	10 µg/L	AG, R	E
2,4-Dimethylphenol	8270	10-µg/L	-AG,-R	, Е
2,4-Dinitrophenol	8270	50 μg/L	AG, R	E
2-Chlorophenol	8270	10 μg/L	AG, R	E
2-Nitrophenol	8270	10 μg/L	AG, R	E
4-Nitrophenol	8270	50 μg/L	AG, R	E
2-Methyl-4,6-dinitrophenol	8270	50 μg/L	AG, R	E
Pentachlorophenol	8270	50 μg/L	AG, R	E
4-Chloro-3-methyphenoi	8270	10 μg/L	AG, R	E
Phenol	8270	10 μg/L	AG, R	E
Acenaphthene	8310	1.0 μg/L	AG, R	. Е
Acenaphthylene	8310	1.0 µg/L	AG, R	E
Anthracene	8310	1.0 µg/L	AG, R	E
Benzo(A) anthracene	8310	1.0 µg/L	AG, R	E
Benzo(A) pyrene	8310	2.0 µg/L	AG, R	E
Benzo(B)fluoranthene	8310	2.0 µg/L	AG, R	E
Benzo(G,H,I)fluoranthene	8310	5.0 µg/L	AG, R	E
Benzo(K)fluoranthene	8310	2.0 µg/L	AG, R	E
Chrysene	8310	1.0 μg/L	AG, R	E
Dibenzo(A,H) anthracene	8310	5.0 μg/L	AG, R	E
Fluoranthene	8310	1.0 µg/L	AG, R	E
Fluorene	8310	1.0 μg/L	AG, R	E

# TABLE 11E-3

# LEACHATE SAMPLING PARAMETERS AND METHODS

Parameter	Analytical Method <sup>1</sup>	Estimated Detection Limit	Container & Preservative	Holding Time
Indeno(1,2,3-CD)pyrene	8310	5.0 µg/L	AG, R	E
Naphthalene	8310	1.0 gg/L	AG, R	E
Phenanthrene	8310	1.0 µg/L	AG, R	E
Pyrene	8310	.1.0 µg/L	AG, A	E

#### NOTES:

- 1 Test Methods for Evaluating Solid Waste," SW-846, Third Edition.
- USEPA Methods 600/4-79/200.
- P Plastic.
- G Glass.
- R Refrigeration.
- N Nitric acid to pH <2.
- T Tefion@-lined cap.
- E 7 days to extraction and 40 days from extraction.
- S Sulfuric acid to pH <2.
- AG Amber glass.
- C Hydrochloric acid to pH <2.
- H NaOH to pH >12.

Some detection limits may not be met for leachate analysis due to matrix interference. In this case, Method 8270 may be used in place of Method 8310. A narrative will be provided by the laboratory to explain any raised detection levels.

		Ford Allen Park Clay Mine Ford Motor Company Leachate
	Date	•
	Time	
	Cell #	
((~))		: -
	Preserved with	
	Samplers Signatures	
	Time	Date

EACH SAMPLE TRANSFERRED SHOULD HAVE A LABEL ATTACHED.

SAMPLE LABEL
FORD MOTOR COMPANY
ALLEN PARK, MICHIGAN



DWN. BY: TBM

APPROVED BY:

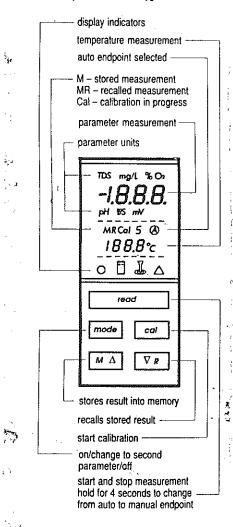
DATE: NOVEMBER 1993 PROJ. 2804.05 FILE # 28040517

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	EQUII	PMENT OPERA	TION AND	CALIBRATION	I MANUALS		
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# M90 Operating Instructions

The M90 is a portable, microprocessor based, pH conductivity and dissolved oxygen meter.

30



# Installing the Battery . . .

Remove rubber battery door on the rear of the meter. Fit battery - alkaline type 9V PP3/1604 or equivalent. Make sure polarity is correct when fitting. The meter must be calibrated after replacing the battery.

#### Installing a Sensor . . .

Locate the pins of the sensor in the meter and push firmly into the meter. The sensor is removed by squeezing down the catch at the rear and pulling the sensor away from the meter.

#### Making a Measurement . . .

pH - remove the sensor wetting cap and slide the vent sleeve to expose the fill hole.

D.O. - remove the sensor wetting cap. Move the sensor in a gentle circular motion when measuring.

Cond - immerse probe to halfway point in solution.

Press mode, read, cal or M to turn meter on and start measurement. Place sensor into solution. Automatic endpoint detection freezes the display when plateau is reached; to manually endpoint press read. Press read again to start new measurement

Continuous measurement may be selected by pressing and holding read for 4 seconds. (In this mode battery consumption is higher and the meter will not automatically switch off when not in use.) Return to auto endpoint by pressing and holding read for 4 seconds.

After use, close the fill hole (pH) and replace the wetting cap (pH and DO).

# Calibrating . . .

For greater accuracy, calibrate the meter regularly.

#### 1 point

Place the sensor in the calibration medium:

	riace the sensor in the callorating medium.			
	pН	pH 7 buffer	7.00 pH (at 25°C	
	Cond	Hold in free air	0.00 μS	
	TDS	Hold in free air	0.00 mg/L	
	DO	Zero oxygen solution	0. %O,	
(mg/L O, is calibrated in %O, mode).				

Press cal - cal 1 is displayed. After endpointing the display automatically updates to the calibrated value shown, or the temperature compensated value.

If read is pressed after Cal 1 update, the meter assumes one point calibration only is required. Samples can now be measured.

#### 2 point

Place the sensor in the second calibration medium: pH pH 4 or 10 buffer 4.00 or 10.01 pH (at 25°C) Cond Cond std. A or B 1413 µS or 12.88 mS TDS Cond std. A or B 706 mg/L or 6.44 g/L DO Hold in air 10 mm above fresh water 100 %O,

Press cal - cal 2 is displayed. After endpointing the display automatically updates to the calibrated value shown or the temperature compensated value.

#### Dissolved oxygen only

In practice, cal 2 is required more frequently than cal 1, this is achieved by pressing cal twice to advance directly to cal 2.

In mg/L O, mode, correction may be made for salinity and barometric pressure. Press cal - 100 is displayed. Use ▲ and ▼ to adjust the display according to these tables:

Cond (mS)	Salinity (g/L)	Setting	
-	0.0	300	
. 6	2.5	97	
11	5.0	95	
15	7.5	<b>9</b> 3	
20	10.0	90	
24	12.5	88	
27	15.0	85	
31	17.5	82	
34	20.0	81	
Barometric	Setting	Barometric	Setting
Pressure (mm)		Pressure (mm)	=
500	79	720	95
600 620 640	82	740	97
£ 640	84	760	100
660	87	780	103
680	90	800	105
700	92	820	108

e.g. if salinity = 5 g/L, setting = 95 if pressure = 740 mm setting = 97 for both parameters, setting will be 95 x 97% = 92.

# Using the Memory ...

5 measurements can be stored in the memory.

# Entering a reading into memory

Press M when measurement has endpointed.

M 1 (or M 2 – M 5 if readings have already been stored) is displayed indicating that the reading has been saved. Flashing M indicates memory is full.

#### Recalling memory

Press R – the last saved measurement is displayed. Press R again to recall the previously saved measurement. MR 1 to MR 5 indicates which saved measurement is being displayed.

#### Clearing last memory

In memory recall mode (MR) press M to clear the measurement being displayed. Only the last entered measurement can be cleared, i.e. if 4 measurements are saved, the M 2 cannot be cleared without first clearing M 4 and M 3.

#### Clearing all memory

Press Runtil M C is displayed, then press M. All memory will be cleared.

# Changing Mode . . .

Press **mode** to enter the second function of the sensor, i.e. pH to mV, conductivity to TDS, %O<sub>2</sub> to mg/L O<sub>2</sub>.

Press mode again to turn the meter off.

# Display Codes/Problem Solving ...

The test plug is provided to test the meter.
Replace the sensor with the test plug and press
read. The display should read:
7.00 pH ± 0.5, 25°C ± 2

tip is immersed in solution and the wetting cap is removed (if applicable)

E2 - cal 1 out of range ) check correct calibration E3 - cal 2 out of range ) medium is used.

Condition/renew sensor

E4 - sensor disconnected

low battery voltage

low electrode slope: condition/renew sensor (pH only)

Default calibration values in memory. Sensor not calibrated (2 point), or battery replaced since last calibration

O manual endpoint selected (continuous measurement)

auto endpoint selected

keypad not responding - replace battery and recalibrate.

# Operating Hints . . .

- Use distilled water when transferring from one solution to another.
- Response time is a function of the sensor and the solution. If the solutions are at different temperatures (or ionic strength – pH only) allow more time for the sensor to respond.
- Avoid handling the sensor tip.
- Make sure no large air bubbles are trapped under the sensor when making measurements.
- Do not use calibration standards after the expiration date.
- Wetting caps should contain: pH – pH 7 buffer; DO – distilled water.
- For greatest accuracy calibrants and samples should be at the same temperature.
- pH keep the electrode filled with the appropriate fill solution to prevent reading drift.
- Conductivity the sensor shield and probe should be kept clean. Make sure no air bubbles are in the cell chamber during measurement.
- 10. Dissolved oxygen -- the sensor can be removed for several hours as a rechargeable battery in the sensor will maintain polarization. For longer periods the sensor should remain connected to the meter to maintain polarization and recharge the sensor battery.

# Reordering Information . . .

Cal.
473617
473619
473620
473621
473622
473623
473624
473625
473626
473650
473651
473652
473676
473654
474594
478540
478570
478510
477269

# Meter Specification . . .

Temperat	lurs compensation: pH Conductivity/TDS Oxygen	0 - 100°C 0 - 50°C 0 - 40°C
Temp pH mV Cond	Range -0.5°C - 100°C 0 - 14 pH 0 - ± 1000 mV 0.00 - 19.99 µS 20.00 µS - 199.9 µS	Resolution 0.1°C 0.01 pH 1 mV 0.01 µS 0.1 µS
TOS	200 – 1999 µS 2.00 – 19.99 mS 0.00 – 10.00 mg/L 10.0 – 100.0 mg/L 100 – 1000 mg/L	1 μS 0.01 mS 0.01 mg/L 0.1 mg/L 1 mg/L
DO	1.00 – 10.00 g/L 0 – 200% O <sub>3</sub> 0 – 20.0 mg/L	0.01 g/L 1% 0.1 mg/L

Auto switch off: if not operated for 10 minutes after endpoint.

Memory: up to 5 measurements can be stored.

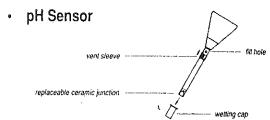
Coming Incorporated Science Products Division Coming, New York 14831 USA Tel: 1-607-737-1667

473655 Rev. A, 3/89

# ੇensor Information

for direct in

id leaded pH, DO and Conductivity Sensors



#### For optimum performance:

- Before use remove wetting cap from tip of sensor, and slide the vent sleeve to expose the fill hole.
- Make sure that the fill solution is not more than 25 mm (1 inch) below the fill hole. Add KCI solution if necessary.
- 3. Gently tap the sensor to remove any air bubbles at the ceramic junction.
- Condition the new sensor by soaking in pH 7 buffer for 2 hours. Prolonged soaking is not recommended.
- Calibrate and measure samples as described in the M90 instructions. Allow sufficient time for the sensor to stabilize when measuring samples of different temperatures, or of low ionic strength. Manual endpointing is advised with these samples.
- After use, check the level of fill solution, reposition the vent sleeve to cover the fill hole, and replace the wetting cap containing pH 7 buffer (if the sensor will not be used again for more than 2 days, we recommend using saturated KCI in the wetting cap).

#### Precautions and Limitations:

- 1. Do not wipe the sensor tip blot dry with a lint-free tissue.
- Do not use KCI saturated with AgCI as this may damage the reference element.
- Do not leave the sensor in organic solvents, strong basic solutions, concentrated fluoride solutions, or hydrofluoric acid for extended periods. Measurements made in these solutions should be taken quickly and the sensor rinsed immediately with distilled water. After rinsing, soak in pH 7 buffer for 2 hours.
- Do not measure solutions that exceed a temperature range of 0 − 100°C.

# Maintenance and Troubleshooting:

Prolonged use and ageing may reduce performance i.e. slow response, low slope values, continuous drift or erratic readings. These may be caused by: Air in junction – remove air bubbles by gentle tapping.

Excess KCl crystals – KCl crystals may build up and settle on the sensor tip, or the KCl may become discolored. Remove the old fill solution and use warm distilled water to dissolve the crystals. Remove water and refill using fresh KCl solution.

Blocked junction – KCl crystals can block the junction. To test for this, blot the tip dry and air dry for one hour. If no KCl crystals appear at the tip of the sensor the junction is blocked. Remove the ceramic junction using tweezers, and insert new junction (Cat. 477269). Tap gently to remove any air bubbles. Contaminated pH bulb – i.e. protein/oil contamination.

Protein - soak the sensor in 10% pepsin solution adjusted to pH 2 with HCl

for 30 immutes. Hinse with restilled water and soak in ph 7 buffer for 2 hours. Oil – wash sensor tip with the second solution. Do not soak the

sensor in acetone solution with distilled water and soc

may cause the seals to deteriorate. Rinse 17 buffer for 2 hours.

Dissolved Oxygen Sensor

replaceable membrane cap ------- wetting cap

#### Installation:

DO membrane caps are fragile. Handle with care to prevent damage. The sensor is shipped dry and must be filled before use. Unscrew the membrane cap from the sensor. If the silver/gold tip is tarnished clean carefully using electrode cleaning compound or silver polish, paying particular attention to the gold cathode. Rinse tip with DO electrolyte, and fill membrane cap, avoiding air bubbles. Hold the sensor vertically and gently screw the membrane cap onto the sensor, allowing surplus electrolyte to run out. Fit sensor to the meter and allow 1 hour minimum for polarization. Calibrate as described in M90 instructions.

#### For optimum performance:

- 1. Before use remove wetting cap from tip of sensor.
- For immediate use the sensor should be kept connected to the meter. The sensor may be removed for up to 3 hours as a rechargeable battery in the sensor will maintain polarization. For extended storage remove the membrane cap and rinse with water, and clean the sensor tip. Store dry with the membrane cap loosely fitted. Do not fit wetting cap.
- When making measurements the sample should be stirred at a constant speed i.e. approximately 20 cm/second (8 inches/second).
- 4. Allow sufficient time for the sensor to stabilize when measuring samples of different temperatures in some cases this can be several minutes. Manual endpointing is advised with these samples. Make sure the sensor is immersed to a depth of at least 40 mm (1.5 inches) to cover the temperature sensing element.
- After use replace wetting cap containing distifled water to prevent electrolyte from drying out.
- Regular maintenance is important to ensure optimum performance.
   Replacement of membrane caps depends on usage we recommend replacement every 2 to 4 weeks.

# Maintenance and Troubleshooting:

If the sensor will not calibrate, or becomes sluggish or erratic:

- The silver/gold sensor tip may become tarnished with time. For optimum
  performance clean tip and refill cap every 2 weeks as described in
  Installation.
- The zero oxygen solution will absorb oxygen if left exposed to air and this will cause inaccurate calibration. Use fresh zero oxygen solution.
- Make sure there are no air bubbles inside the membrane cap when filling with DO electrolyte. Check by looking up through the membrane from the bottom of the sensor.
- Check the membrane for damage and replace with new cap (Cat. 473626) as necessary.

Conductivity Senso

#### For optimum performance:

- 1. Make sure the clear plastic shield is in place when measurin
- When measuring make sure the solution is above the cell ch and below the vent hole.
- To prevent carryover from high to low conductivity solutions distilled water between measurements.
- Make sure the cell chamber is bubble free when measuring, bubbles, immerse probe in the solution at an angle and then vertical position.
- Allow sufficient time for the sensor to stabilize when measuri different temperatures. Manual endpointing is advised with the
- The sensor is not recommended for low ionic strength solution
- Clean the probe and shield with distilled water after use.

# General Troubleshooting for all Sensors

- To verify meter is working check using the test plug.
- If the sensor connector becomes damaged or wet the display E4 when a sensor is connected.
- If the temperature sensing element becomes damaged the te display may read E1 when a sensor is connected.

# **Ordering Information:**

item pH sensor pH electros

pH electrode fill solution, 3 x 5 mL

pH 7 buffer sachet (pack of 30)

pH 4 buffer sachet (pack of 30) pH 10 buffer sachet (pack of 30)

pH multipack, pH 4, 7 and 10 (pack of 30 assorted sachets)

Buffer solution pH 4.00, 2 x 500 mL (red) Buffer solution pH 7.00, 2 x 500 mL (yellow)

Buffer solution pH 10.01, 2 x 500 mL (blue)

Buffer rainbow pack, pH 4.00, 7.00 and 10.01 (2 x 500 mL of each Replaceable ceramic junctions (pH), pack of 3

DO sensor

DO electrolyte, 3 x 5 mL Zero oxygen solution, 500 mL

DO membrane replacement kit, pack of 2

p0, electrode cleaning compound

Conductivity/TDS sensor

1413 µS conductivity standard, 500 mL 12.88 mS conductivity standard, 500 mL

Rinse solution sachet (pack of 30)

Corning Incorporated Science Products Division Corning, New York 14831 USA Tel: 1-607-737-1667

Technical Information Center: 1-(800)-222-7740

# ATTACHMENT 11F SURFACE WATER MONITORING PLAN AND PROCEDURES

### **ATTACHMENT 11F**

# **Surface Water Monitoring Plan and Procedures**

A surface water sample will be collected on a quarterly basis after the initiation of filling in Cell II. The surface water analytical parameter list and sample collection procedures are discussed below. Proposed additional background surface water sampling, to be carried out every other month for a period of 1 year, is also discussed below. Statistical procedures for evaluating data are presented in Subsection 11F.3. A detailed laboratory QA/QC program is provided in Attachment 11A.

# 11F.1 Surface Water Sampling Parameters and Schedule

# Additional Background Surface Water Sampling

The parameters for the proposed additional background sampling include all of the leachate monitoring parameters for which surface water background values have not already been established. Table 11F-1 identifies which of these parameters background soil sampling is completed for, and which parameters are proposed for additional background sampling. The additional background sampling will be carried out every other month for a period of 1 year, or after a rainfall event greater than one-half inch until sufficient data is collected, and will consist of collecting an individual sample from Allen Drain, at the point indicated on Figures 11F-1 and 11F-2. If sufficient volume of water is unavailable in the drain, then the sample will be collected from the alternative sampling point, which is located immediately adjacent to the outfall in the sedimentation basin (see Figure 11F-2). In order to maintain consistency with previously collected surface water sampling data, the sample identification code Allen Drain - #-BG will be used to identify additional background surface water samples. Sample volumes, preservation methods, detection limits, holding times, and analytical methods are presented in Table 11F-3. Field and sample handling procedures are discussed below.

### Operational Surface Water Sampling Plan

Operational surface water sampling will consist of collecting an individual sample on a quarterly basis from Allen drain, at the point indicated on Figures 11F-1 and 11F-2. If sufficient volume of water is unavailable in the drain, then the sample will be collected from the alternative sampling point, which is located immediately adjacent to the outfall in the sedimentation basin (see Figure 11F-2). In order to maintain consistency with previously collected surface water sampling data, the sample identification code Allen Drain -#-OP will be

used to identify the operational surface water sample. The parameter list for operational surface water sampling is presented in Table 11F-2. Sample preservation methods, detection limits, holding times, and analytical methods are presented in Table 11F-3. The operational monitoring parameter list (Table 11F-2) will be reviewed annually and revised if appropriate, as described in Subsection 11.3.2 of this application and in Attachment 11E, the Leachate Sampling and Monitoring Plan.

# 11F.2 Sample Collection Procedures

- The procedures for surface water sample collection will be as follows:

- Sample collection bottles will be provided pre-preserved from the analytical laboratory. Prepare bottles by labeling according to EPA Chain-of-Custody (COC) requirements using a sample tag as shown on Figure 11F-3, or equivalent. The tag will be filled out in ink and in legible handwriting. To maintain consistency with the historical record of background samples, sample identification code Allen Drain -#-OP or Allen Drain -#-BG, corresponding to an operating plan or background sample, respectively, will be used.
- Spread a new piece of plastic sheeting on the ground next to the surface water sampling location in order to prevent sampling equipment from contacting the ground. A new pair of disposable PVC or latex gloves will be used by the sampler at each sampling location in order to prevent contamination of the samples.
- 3. Collect samples using a stainless steel dipper, disposable bailer, pre-cleaned Teflon bailer, a dedicated bailer, or laboratory prepared jars.
  - a) If a disposable bailer is used, a new disposable bailer will be used for each sampling event, and a new piece of nylon rope will be used to lower the bailer into the surface water body.
  - b) If a pre-cleaned bailer is used, the bailer will be precleaned prior to the sampling event according to the following procedures:
    - Brush with soapy (phosphate free soap) water and soak for a minimum of 4 hours
    - . Rinse with potable water for 3 minutes
    - Rinse with 10 percent nitric acid solution
    - Rinse with deionized water
    - Oven dry
    - . Seal in polypropylene plastic to prevent contamination prior to use
  - c) If a dedicated bailer is used, the surface water sampling point will have a bailer dedicated for use only at that sampling point. The bailer will be labeled and stored in a clean, plastic bag in an indoor storage location while not in use. The bailer will be decontaminated prior to and after use by washing with soapy (phosphate-free soap) water and

- double rinsing with deionized water. A new piece of nylon rope will be used to lower the bailer into the surface water body.
- d) If a stainless steel dipper is used to collect a surface water sample, the dipper will be labeled and stored in a clean, plastic bag in an indoor storage location while not in use. The bailer will be decontaminated prior to and after use by washing with soapy (phosphate-free soap) water and double rinsing with deionized water.
- e) If laboratory prepared jars are used, no preservative or fixative will be used.
- Sample vials for volatile organics should be filled first. The sample should be collected in a manner which minimizes sample disturbance (i.e., slowly lower the bailer or dipper into the surface water body with minimal disturbance). The surface water stream should be allowed to strike the inner wall of the vial to minimize formation of air bubbles. Fill the sample vial or bottle with a minimum of splashing. Fill each vial until the water forms a positive meniscus at the brim. Allow the vial to overflow slightly, then replace the cap by gently setting it on the water meniscus. Tighten firmly, but do not over-tighten.
- 5. Invert the vial and tap lightly to check for air bubbles.
- 6. Place samples on ice immediately.
- 7. Fill a clean plastic bucket with surface water and allow to settle for 5 minutes. Decant water into appropriate bottles for other sample parameters. Note that surface water samples are not field filtered for any analytical parameters. Place samples in coolers with ice or a refrigerator immediately following sample collection. All samples will be stored in ice coolers from time of collection through delivery to the analytical laboratory. Decon the bucket by brushing with soapy (phosphate-free) water and rinsing three times with potable water.
- 8. Fill a disposable plastic sample cup with 200 to 300 mL of surface water and collect temperature, pH and specific conductance measurements. These measurements will be collected with a Corning Model M90 pH and conductivity meter, or equivalent, according to the manufacturer's specifications. The pH and conductivity probes will be cleaned between sampling locations by double rinsing with distilled water. The Model M90 meter will be calibrated once prior to use for every 4 hours of use in the field, according to the manufacturer's specifications for calibration. Manufacturer's specifications for instrument use and calibration are included at the end of this Attachment.
- 9. Field measurements will be recorded on field data sheets, which will identify the sampling location, time, date, and sampler, along with the measurements collected and a description of the number and type of sample bottles filled. Copies of the field data sheets will be retained at the facility.

10. The Chain-of-Custody (COC) Form describing the sample will be drawn up by the sampler. This form will accompany all samples, and be signed and dated by the sampler at the time the samples are released. The individual accepting the samples will also sign and date the same COC form acknowledging receipt of the samples. Copies of the COC will be retained at the facility.

### 11F.3 Statistical Evaluation of Surface Water Monitoring Data

The results of the quarterly sampling to be conducted at surface water monitoring point will be compared against the background data set for the following parameters: chrome, copper, arsenic, selenium, volatile and semi-volatile organic compounds. This list may be revised on an annual basis as described in Attachment B. The following procedures will be used to evaluate each parameter listed in the statistical evaluation program. If comparison of any of the operational monitoring data to background data results in a statistically significant difference, the steps outlined in Subsection 11.6.3 will be followed.

- Tabulate, evaluate, and reduce the existing background data. Review data to determine completeness and to determine if sample locations were consistent and clearly defined. Review any additional background data in the same manner when background data collection is complete.
- 2. Revise, if necessary, the estimated quantitation limits (RDLs) for each constituent. RDLs reported by analytical laboratories may change with time. Use the largest detection limit for each parameter wherever a correction for censored data is needed. Using the largest reported RDL value for each constituent is appropriate because the maximum value sets the level of accuracy that can be attained in future monitoring, even if RDL values decrease in the future.
- 3. If the background data set is 100 percent censored, do not perform statistics. The actual level of detection will serve as the trigger value for resampling. In this case, an operational monitoring sample result which exceeds the analytical detection limit will be confirmed by collecting an individual sample at that location and analyzing for the parameter which exceeded background. This measure is being taken in order to rule out laboratory error as a source of the detection. If the analyte is not detected in the confirmatory sample, no further action will be taken. If the analyte is detected, resample at that location in quadruplicate. If any of the four reported concentrations exceed the detection limit, then follow the procedures in Subsection 11.6.3.
- 4. Assess the underlying statistical distribution of the data, and correct for log normality if necessary. After the first round of statistical evaluation has been completed, this step will consist of transforming the current data, if necessary, based on the previous evaluation. As recommended in the February, 1993 USEPA Guidance document, assess normality by constructing probability plots.

Follow the attached guidance for constructing probability plots. The plotted points will approximate a straight line if the data are normal. Construct probability plots of the log-transformed and the raw data for each parameter. Compare the plots and decide which representation of the data is closer to the normal distribution. If the log transform of the data is selected as an appropriate transformation, transform all background and operational data for that sample point for that parameter prior to conducting any statistical tests on the data. State whether the statistical test was conducted on raw or transformed data in all reports regarding the data.

- 5. Inspect the data set for outliers. Conduct formal testing for outliers only if a reported concentration is orders of magnitude higher than the rest of the data set. Follow the procedure for outlier testing presented at the end of this attachment. The outlier test assumes that the data other than the outlier follows a normal distribution. Therefore, if the data set is log-normally distributed (see number 3 above), conduct the outlier test on the log-transformed data. Correct or remove an outlier from the data set only if the value can be identified as:
  - (1) an error in transcription or dilution;
  - (2) a documented error in an analytical procedure or report of matrix interferences in the procedure; or
  - (3) some other factor from those listed in the RCRA guidance (USEPA, 1989; USEPA, 1993).

In the event an outlier can be verified, obtain MDNR permission before removing the outlier from the data set. If no obvious cause can be identified for a value being an outlier, it will remain in the operational data set used for statistical evaluation unless MDNR approval is obtained to remove it.

- 6. Inspect the current round of data for nondetects. If a parameter was reported to be below the RDL for that round, then do not perform a statistical test with that data (i.e., do not perform a statistical evaluation to determine if a nondetect represents an exceedance of background). Add the analytical result to the database for that sample point. This approach is being taken because it is reasonable to assume that a nondetect cannot represent an exceedance of background.
- 7. Evaluate the degree of censorship in the data, and select the appropriate statistical test based on this evaluation. After the first round of statistical evaluation has been completed, this step will consist of evaluating whether the statistical test used during previous rounds remains an appropriate choice for the data set. The following steps will be followed in determining how censored data will be handled and in choosing the statistical test to be performed for each sampling point:
  - a) If the percentage of nondetects in the database for the sample point is less than 15 percent, substitute of a value of 1/2 the RDL for all nondetects and calculate a prediction interval according to the procedure included at the end of this Attachment.

- b) If the percentage of nondetects in the database for the sample point is between 15 and 50 percent, use Cohen's or Aichison's adjustment to calculate the mean and standard deviation of the background data. Use these adjusted statistics to calculate a prediction interval. Follow the procedures presented at the end of this attachment to determine which of Cohen's or Aichison's adjustment should be used. Calculate the prediction interval according to the presented procedure.
- c) If the percentage of nondetects in the database for the sample point is between 50 and 90 percent, use the Wilcoxan Rank-Sum Test to compare operational monitoring results to background data. Procedures are attached.
- d) If the percentage of nondetects in the database for the sample point is 90 percent or greater, calculate a Poisson prediction limit. Procedures are included at the end of this Attachment.
- e) If the percentage of nondetects in the background data set is 100 percent follow the procedure listed above in step number 3.
- 8. If the statistical test performed for step number 7 above indicates that an exceedance has occurred, follow the steps outlined in Subsection 11.6.3 of the Permit.

### TABLE 11F-1

BACKGROUND SURFACE WATER SAMPLING PARAMETERS			
Parameters for	which Surface Water Background Sampling	) is Completed:	
Alkalinity (as CaCo <sub>3</sub> ) Ammonia - N Arsenic Barium Bicarbonate alkalinity BOD Cadmium Calcium Carbonate alkalinity Chloride  2-Chlorophenol 4-Chloro-3-methylphenol 2,4-Dichlorophenol	Chromium COD Copper Cyanide Iron Lead Magnesium Mercury Nickel Nitrate - N	pH (field and laboratory) Selenium Silver Sodium Specific conductance (field and laboratory) Sulfate TOC Total recoverable phenolics Zinc  Phenol 2,4,5-Trichlorophenol 2,4,6-Trichlorophenol	
2,4-Dinitrophenol 2,4-Dinitrophenol Scal	Pentachlorophenol  7 Polynuclear Aromatic Hydrocarbons (PA	NHs):	
Acenaphthene Acenaphthylene Anthracene Benzo(A) anthracene Benzo(A) pyrene Benzo(B) fluoroanthene	Benzo(G,H,I) perylene Benzo(K)fluoranthene Chrysene Dibenzo(A,H) anthracene Fluoranthene Fluorene	Indeno (1,2,3-CD) pyrene Naphthalene Phenanthrene Pyrene	
Proposed Ad	ditional Background Surface Water Samplin	g Parameters	
Antimony Beryllium Cobalt Thallium	Tin Vanadium		
	Volatile Organic Compounds (VOCs):		
Benzene Bromodichloromethane Bromoform Bromomethane Carbon tetrachioride Chlorobenzene Chloroethane 2-Chloroethylvinyl ether Chloroform Chloromethane Dibromochloromethane	1,2-Dichlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene Dichlorodifluoromethane 1,1-Dichloroethane 1,2-Dichloroethene trans-1,2-Dichloroethene 1,2-Dichloropropane cis-1,3-Dichloropropene trans-1,3-Dichloropropene	Ethylbenzene Methylene chloride 1,1,2,2-Tetrachloroethane Tetrachloroethene Toluene 1,1,1-Trichloroethane 1,1,2-Trichloroethane Trichloroethene Trichloroethene Trichlorofluoromethane Vinyl chloride	

### NOTES:

- Surface water samples are analyzed for total metals and are therefore not field-filtered. 1.
- 2. VOCs include purgeable halocarbons and aromatics listed in Appendix A to Part 136 (40 CFR) - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater.

TABLE 11F-2				
SURFACE WAT	SURFACE WATER OPERATIONAL MONITORING PARAMETERS			
	Total Metals	Y		
Antimony Arsenic Barium Beryllium Cadmium Chromium	Cobalt Copper Lead Mercury Nickel Selenium	Silver Thallium Tin Vanadium Zinc		
	Inorganics and Other Parameters			
Bicarbonate alkalinity Carbonate alkalinity Calcium Chloride	Cyanide Iron Magnesium pH*	Sodium Specific conductance* Sulfate Total recoverable phenolics		
Scar	n 7 Polynuclear Aromatic Hydrocar	bons		
Acenaphthene Acenaphthylene Anthracene Benzo(A)anthracene Benzo(A)pyrene Benzo(B)fluoroanthene	Benzo(G,H,I)perylene Benzo(K)fluoranthene Chrysene Dibenzo(A,H)anthracene Fluoranthene Fluorene	Indeno(1,2,3-CD)pyrene Naphthalene Phenanthrene Pyrene		
V	olatile Organic Compounds (VOCs	)		
Benzene Bromodichloromethane Bromoform Bromomethane Carbon tetrachloride Chlorobenzene Chloroethane 2-Chloroethylvinyl ether Chloroform Chloromethane Dibromochloromethane	1,2-Dichlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene Dichlorodifluoromethane 1,1-Dichloroethane 1,2-Dichloroethene trans-1,2-Dichloroethene 1,2-Dichloropropane cis-1,3-Dichloropropene trans-1,3-Dichloropropene	Ethylbenzene Methylene chloride 1,1,2,2-Tetrachloroethane Tetrachloroethene Toluene 1,1,1-Trichloroethane 1,1,2-Trichloroethane Trichloroethene Trichlorofluoromethane Vinyl chloride		

### NOTES:

- \* Specific conductance and pH are measured in the field.
- 1. Surface water samples are analyzed for total metals and are therefore not field-filtered.
- VOCs include purgeable halocarbons and aromatics listed in Appendix A to Part 136 (40 CFR) - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater.
- 3. This list will be reviewed annually and revised if appropriate as discussed in Subsection 11.6.2.

TABLE 11F-3
SURFACE WATER SAMPLING PARAMETERS AND METHODS

Parameter	Analytical Method <sup>1</sup>	Estimated Detection Limit	Container & Preservative	Holding Time
Antimony	6020	1 μg/L	G, N	6 months
Arsenic	6020, 7061	1 μg/L	G, N	6 months
Barium	6020	5 μg/L	G, N	6 months
Beryllium	-6020	1-μg/L	· G;+N · ·	6 months
Cadmium	6020	0.2 µg/L	G, N	6 months
Chromium	6020	1 μg/L	G, N	6 months
Cobalt	6010	15 μg/L	G, N	6 months
Copper	6010	1 μg/L	G, N	6 months
Lead	6020	1 μg/L	G, N	6 months
Mercury	7470	0.2 μg/L	G, N	38 days
Nickel	6020	50 μg/L	G, N	6 months
Selenium	6020, 7741	1 μg/L	G, N	6 months
Silver	6020	0.5 μg/L	G, N	6 months
Thallium	6020	2.0 µg/∟	G, N	6 months
Tin	200.7 <sup>2</sup>	500 μg/L	G, N	6 months
Vanadium	6010	10 μg/L	G, N	6 months
Zinc	6020	4 μg/L	G, N	6 months
Iron	6010	0.02 mg/L	G, N	6 months
Bicarbonate alkalinity	310.1	5 mg/L	P, R	14 days
Carbonate alkalinity	310.1	10 mg/L	P, R	14 days
Calcium	6010	1 mg/L	G, N	6 months
Cyanide	335.2	5 μg/L	P, H	14 days
Magnesium	6010	1 mg/L	G, N	6 months
Sodium	6010	1 mg/L	G, N	6 months
Chloride	35.2	1 mg/L	P, R	28 days
Sulfate	375.4	2 mg/L	P, R	28 days
Total recoverable phenolics	9065	0.01 mg/L	AG, T, S	28 days
Benzene	8260	1.0 mg/L	C, G, T, R	14 days
Ethylbenzene	8260	1.0 mg/L	C, G, T, R	14 days
Toluene	8260	1.0 mg/L	C, G, T, R	14 days

June 1995

TABLE 11F-3
SURFACE WATER SAMPLING PARAMETERS AND METHODS

Parameter	Analytical Method <sup>1</sup>	Estimated Detection Limit	Container & Preservative	Holding Time
Vinyl chloride	8260	5.0 mg/L	C, G, T, R	14 days
Bromodichloromethane	8260	1.0 mg/L	C, G, T, R	14 days
Bromoform	8260	1.0 mg/L	C, G, T, A	14 days
Bromomethane	. 8260	5.0 .mg/L	_C, G, T, R	14 days
Carbon tetrachloride	8260	1.0 mg/L	C, G, T, R	14 days
Chlorobenzene	8260	1.0 mg/L	C, G, T, R	14 days
Chloroethane	8260	5.0 mg/L	C, G, T, R	14 days
2-Chloroethylvinyl ether	8260	5.0 mg/L	C, G, T, R	14 days
Chloroform	8260	1.0 mg/L	C, G, T, R	14 days
Chloromethane	8260	5.0 mg/L	C, G, T, A	14 days
Dibromochloromethane	8260	1.0 mg/L	C, G, T, A	14 days
1,2-Dichlorobenzene	8260	1.0 mg/L	C, G, T, A	14 days
1,3-Dichlorobenzene	8260	1.0 mg/L	C, G, T, R	14 days
1,4-Dichlorobenzene	8260	1.0 mg/L	C, G, T, A	14 days
Dichlorodifluoromethane	8260	5.0 mg/L	C, G, T, R	14 days
1,1-Dichloroethane	8260	1.0 mg/L	C, G, T, R	14 days
1,2-Dichloroethane	8260	1.0 mg/L	C, G, T, R	14 days
1,1-Dichloroethene	8260	1.0 mg/L	C, G, T, R	14 days
trans-1,2-Dichloroethene	8260	1.0 mg/L	C, G, T, R	14 days
1,2-Dichloropropane	8260	1.0 mg/L	C, G, T, R	14 days
cis-1,3-Dichloropropene	8260	1.0 mg/L	C, G, T, R	14 days
trans-1,3-Dichloropropene	8260	1.0 mg/L	C, G, T, R	14 days
Methylene chloride	8260	5.0 mg/L	C, G, T, R	14 days
1,1,2,2-Tetrachloroethene	8260	1.0 mg/L	C, G, T, R	14 days
Tetrachioroethene	8260	1.0 mg/L	C, G, T, R	14 days
1,1,1-Trichloroethane	8260	1.0 mg/L	C, G, T, R	14 days
1,1,2-Trichloroethane	8260	1.0 mg/L	C, G, T, R	14 days
Trichloroethene	8260	1.0 mg/L	C, G, T, R	14 days

TABLE 11F-3
SURFACE WATER SAMPLING PARAMETERS AND METHODS

Parameter	Analytical Method <sup>1</sup>	Estimated Detection Limit	Container & Preservative	Holding Time
2,4,5-Trichlorophenol	8270	10 µg/L	AG, R	· E
2,6,4-Trichlorophenol	8270	10 µg/L	AG, R	E
2,4-Dichlorophenol	8270	10 μg/L	AG, R	E
2,4-Dimethylphenol	-8270	10 μg/L	-AG, R	E
2,4-Dinitrophenol	8270	50 μg/L	AG, R	E
2-Chlorophenoi	8270	10 μg/L	AG, R	E
2-Nitrophenol	8270	10 μg/L	AG, R	E
4-Nitrophenol	8270	50 μg/L	AG, R	E
2-Methyl-4,6-dinitrophenol	8270	50 μg/L	AG, R	E
Pentachlorophenol	8270	50 μg/L	AG, R	Ε
4-Chloro-3-methylphenoi	8270	10 μg/L	AG, R	E
Phenol	8270	10 μg/L	AG, R	E
Acenaphthene	8310	1.0 µg/L	AG, R	E
Acenaphthylene	8310	1.0 μg/L	AG, R	E
Anthracene	8310	1.0 μg/L	AG, R	E
Benzo(A) anthracene	8310	1.0 µg/L	AG, R	E
Benzo(A) pyrene	8310	2.0 μg/L	AG, R	E
Benzo(B)fluoranthene	8310	2.0 µg/L	AG, R	E
Benzo(G,H,I)fluoranthene	8310	5.0 μg/L	AG, R	Ε
Benzo(K)fluroanthene	8310	2.0 μg/L	AG, R	E
Chrysene	8310	1.0 μg/L	AG, R	E
Dibenzo(A,H)anthracene	8310	5.0 µg/∟	AG, R	E
Fluoranthene	8310	1.0 µg/L	AG, R	E .
Fluorene	8310	1.0 μg/L	AG, R	E

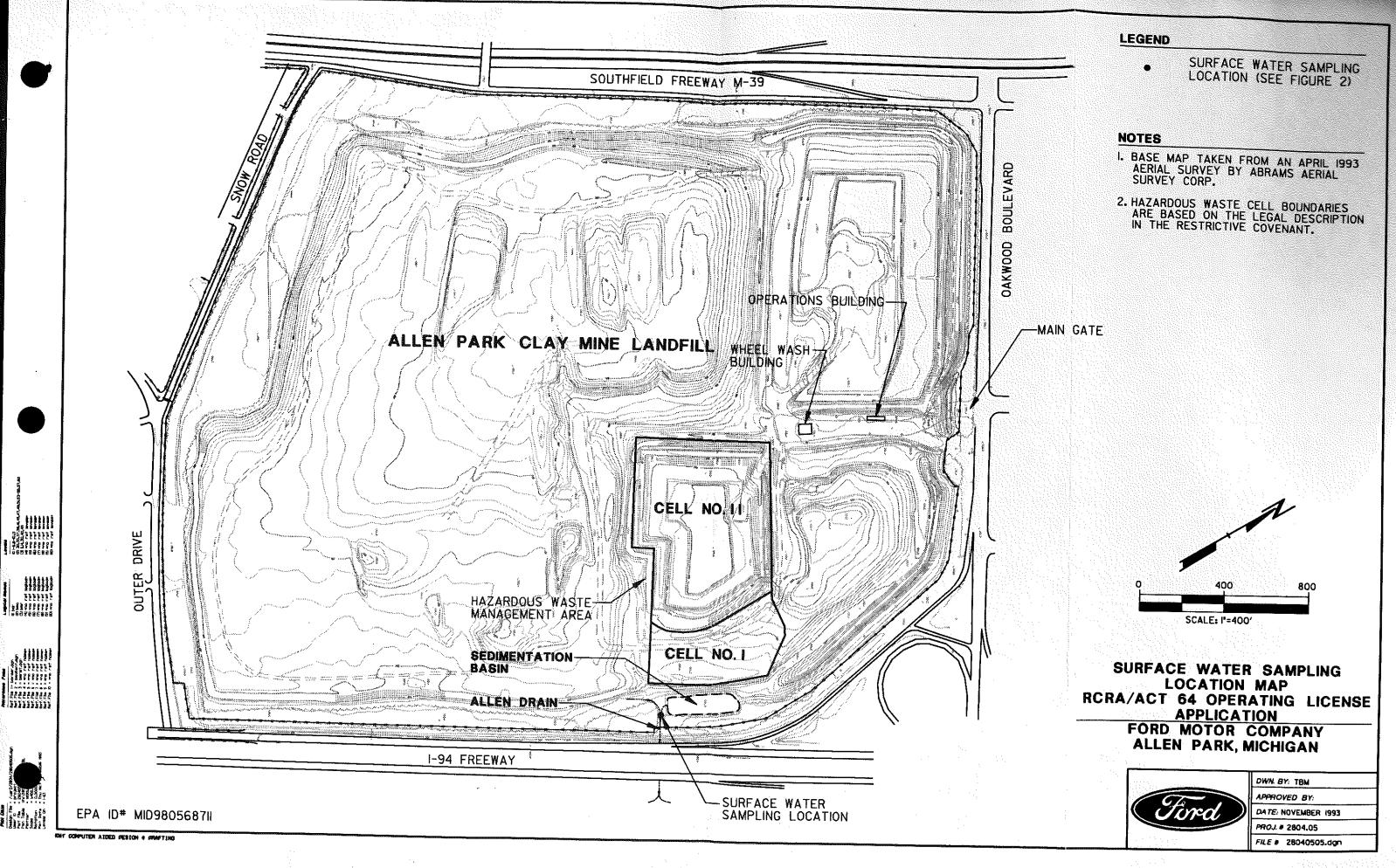
**TABLE 11F-3** 

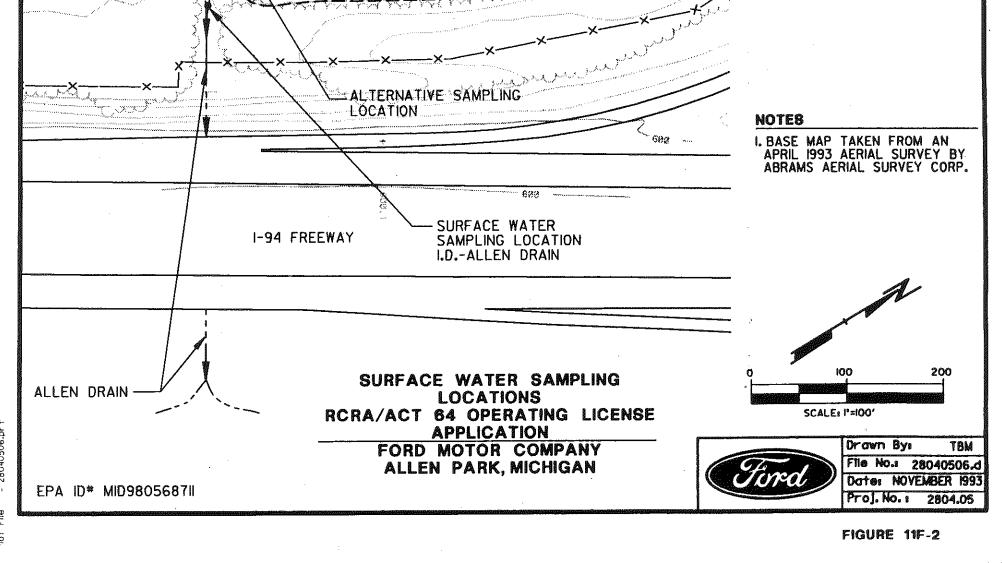
### SURFACE WATER SAMPLING PARAMETERS AND METHODS

Parameter	Analytical Method¹	Estimated Detection Limit	Container & Preservative	Holding Time
Indeno(1,2,3-CD)pyrene	8310	5.0 μg/L	AG, R	Ë
Naphthalene	8310	1.0 µg/L	AG, R	Е
Phenanthrene	8310	1.0 µg/L	AG, R	E
Pyrene	8310	.1.0-µg/L	AG,-R	E

### NOTES:

- "Test Methods for Evaluating Solid Waste," SW-846, Third Edition.
- 2 USEPA Methods 600/4-79/200.
- Plastic.
- G Glass.
- R Refrigeration.
- N T Nitric acid to pH <2.
- Teflon®-lined cap.
- E 7 days to extraction and 40 days from extraction.
- s Sulfuric acid to pH <2.
- AG Amber glass.
- С Hydrochloric acid to pH <2.
- NaOH to pH >12.





SEDIMENTATION BASIN LEGEND

SURFACE WATER

**SAMPLING LOCATION** 

		Ford Allen Park Clay Mins Ford Motor Company Surface Water Allen Drain
-	Dote	
	Time	
	Analyze for	
	Preserved with	
// \	Samplers Signatures	NATIONAL PROPERTY OF THE PROPE
//	Time	Date

EACH SAMPLE TRANSFERRED SHOULD HAVE A LABEL ATTACHED.

SAMPLE LABEL
FORD MOTOR COMPANY
ALLEN PARK, MICHIGAN



DWN. BY: TBM

APPROVED BY:

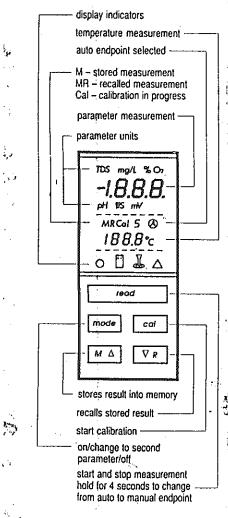
DATE: NOVEMBER 1993

PROJ. # 2804.05
FILE # 28040520

**EQUIPMENT OPERATION AND CALIBRATION MANUALS** 

### M90 Operating Instructions

The M90 is a portable, microprocessor based, pH. conductivity and dissolved oxygen meter.



, 1

### Installing the Battery . . .

Remove rubber battery door on the rear of the meter. Fit battery - alkaline type 9V PP3/1604 or equivalent. Make sure polarity is correct when fitting. The meter must be calibrated after replacing the battery.

### Installing a Sensor . . .

Locate the pins of the sensor in the meter and push firmly into the meter. The sensor is removed by squeezing down the catch at the rear and pulling the sensor away from the meter.

### Making a Measurement . . .

pH - remove the sensor wetting cap and slide the vent sleeve to expose the fill hole.

D.O. - remove the sensor wetting cap. Move the sensor in a gentle circular motion when measuring.

Cond - immerse probe to halfway point in solution.

Press mode, read, call or M to turn meter on and start measurement. Place sensor into solution. Automatic endpoint detection freezes the display when plateau is reached; to manually endpoint press read. Press read again to start new measurement.

Continuous measurement may be selected by pressing and holding read for 4 seconds. (In this mode battery consumption is higher and the meter will not automatically switch off when not in use.)

Return to auto endpoint by pressing and holding read for 4 seconds.

After use, close the fill hole (pH) and replace the wetting cap (pH and DO).

### Calibrating ...

For greater accuracy, calibrate the meter regularly.

### 1 point

		sensor in the calibration	ig medium:
, ,	рΗ	pH 7 buffer	7.00 pH (at 25°C)
- /	Cond	Hold in free air	0.00 μS
•	TDS	Hold in free air	0.00 mg/L
	DO	Zero oxygen solution	0. %O.
;	(mg/L O,	is calibrated in %O, mo	ode).

Press cal - cal 1 is displayed. After endpointing the display automatically updates to the calibrated value shown, or the temperature compensated value.

If read is pressed after Cal 1 update, the meter assumes one point calibration only is required. Samples can now be measured.

### 2 point

Place the sensor in the second calibration medium: pH pH 4 or 10 buffer 4.00 or 10.01 pH (at 25°C) Cond Cond std. A or B 1413 µS or 12.88 mS TDS Cond std. A or B 706 mg/L or 6.44 g/L DO Hold in air 10 mm above fresh water 100 %O,

Press cal - cal 2 is displayed. After endpointing the display automatically updates to the calibrated value shown or the temperature compensated value.

#### Dissolved oxygen only

In practice, cal 2 is required more frequently than cal 1, this is achieved by pressing cal twice to advance directly to cal 2.

In mo/L.O. mode, correction may be made for salinity and barometric pressure. Press cal - 100 is displayed. Use ▲ and ▼ to adjust the display according to these tables:

Cond (mS)	Salinity (g/L)	Setting	
-	0.0	100	
. 6	2.5	97	
11	5.0	95	
15	7.5	93	
20	10.0	90	
24	12.5	88	
27	15.0	85	
31	17.5	82	
34	20.0	81	
Barometric	Setting	Barometric	Setting
Pressure (mm)	•	Pressure (mm)	
5: 600	79	720	95
<sup>تا</sup> 620	82	740	97
- 640	84	760	100
- 660	87	780	103
680	90	800	105
y 700	92	820	108
	6 11 15 20 24 27 31 34  Barometric Pressure (mm) 4 520 4 640 660	- 0.0 6 2.5 11 5.0 115 7.5 20 10.0 24 12.5 27 15.0 31 17.5 34 20.0  Barometric Setting Pressure (mm) 600 79 620 62 640 84 660 87 680 90	- 0.0 100 6 2.5 97 111 5.0 95 115 7.5 93 20 10.0 90 24 12.5 88 27 15.0 85 31 17.5 82 34 20.0 81  Barometric Setting Barometric Pressure (mm) 7 600 79 720 600 82 740 640 84 760 680 90 80

e.g. if salinity = 5 g/L, setting = 95 il pressure = 740 mm setting = 97 for both parameters, setting will be 95 x 97% = 92. Using the Memory ...

5 measurements can be stored in the memory.

Entering a reading into memory

Press M when measurement has endpointed.

M 1 (or M 2 – M 5 if readings have already been stored) is displayed indicating that the reading has been saved. Flashing M indicates memory is full.

Recalling memory

Press R - the last saved measurement is displayed. Press R again to recall the previously saved measurement. MR 1 to MR 5 indicates which saved measurement is being displayed.

Clearing last memory

In memory recall mode (MR) press M to clear the measurement being displayed. Only the last entered measurement can be cleared, i.e. if 4 measurements are saved, the M 2 cannot be cleared without first clearing M 4 and M 3.

Clearing all memory

Press Runtil M C is displayed, then press M. All memory will be cleared.

Changing Mode ...

Press **mode** to enter the second function of the sensor, i.e. pH to mV, conductivity to TDS, %O, to mg/L O,.

Press mode again to turn the meter off.

### Display Codes/Problem Solving ... Using the Test Plug

The test plug is provided to test the meter. Replace the sensor with the test plug and press read. The display should read: 7.00 pH ± 0.5, 25°C ± 2

- E1 measurement out of range: check that sensor & tip is immersed in solution and the wetting cap is removed (if applicable)
- E2 cal 1 out of range | check correct calibration
- E3 cal 2 out of range | medium is used.

-. Couditionalettew a

E4 - sensor disconnected

low battery voltage

low electrode slope: condition/renew sensor (pH only)

Default calibration values in memory. Sensor not calibrated (2 point), or battery replaced since last calibration

- O manual endpoint selected (continuous measurement)
- auto endpoint selected

keypad not responding - replace battery and recalibrate.

### Operating Hints ...

- Use distilled water when transferring from one solution to another.
- Response time is a function of the sensor and the solution. If the solutions are at different temperatures (or ionic strength – pH only) allow more time for the sensor to respond.
- 3. Avoid handling the sensor tip.
- Make sure no large air bubbles are trapped under the sensor when making measurements.
- Do not use calibration standards after the expiration date.
- Wetting caps should contain: pH – pH 7 buffer; DO – distilled water.
- 7. For greatest accuracy calibrants and samples should be at the same temperature.
- pH keep the electrode filled with the appropriate fill solution to prevent reading drift.
- Conductivity the sensor shield and probe should be kept clean. Make sure no air bubbles are in the cell chamber during measurement.
- 10. Dissolved oxygen the sensor can be removed for several hours as a rechargeable battery in the sensor will maintain polarization. For longer periods the sensor should remain connected to the meter to maintain polarization and recharge the sensor battery.

### Reordering Information . . .

	Cal.
Item	473617
Meter only	473619
pH sensor	473620
DO sensor	473621
Conductivity/TDS sensor	473622
Hard carrying case	473623
1413 µS conductivity standard (A), 500 mL	473624
12 BB mS conductivity standard (B), 500 mc	473625
Zero oxygen solution, 500 mL	473626
DO membrane replacement kit	
pH 7 buffer sachet (pack of 30)	473650
pH 4 buffer sachet (pack of 30)	473651
pH 10 buller sachet (pack of 30)	473652
pH multipack, pH 4, 7, 10, (pack of 30 assorted)	473676
pH electrode fill solution, 3 x 5 mL	473654
DO electrolyte, 3 x 5 mL	474594
Butter solution pH 4.00, 2 x 500 mL (red)	478540
Butter solution pH 7.00, 2 x 500 mL (yellow)	478570
Butter solution pri 7.00, 2 x 500 ml (blue)	478510
Buffer solution pH 10.01, 2 x 500 mL (blue) Replaceable ceramic junctions (pH), pack of 3	477269
h Lichiacogolo commo faritamente de 111	

### Meter Specification ...

0 - 200% O,

0 = 20.0 mg/L

Temperature compensation:

pH 0 - 100°C
Conductivity/TDS 0 - 50°C

0 - 40°C Oxygen Resolution Range -0.5°C - 100°C D.1°C Temp 0.01 pH 0 - 14 pH  $0-\pm 1000~\text{mV}$ 1 mV m۷ 0.01 µS 0.00 - 19.99 µS 0.1 µS 20.00 μS - 199.9 μS 1μS 200 - 1999 µS 0.01 mS 2.00 - 19.99 mS 0.01 mg/L 0,00 - 10.00 mg/L 10.0 - 100.0 mg/L 0.1 mg/L 1 mg/L 100 - 1000 mg/L 0.01 g/L 1,00 - 10.00 g/L

Auto switch off: if not operated for 10 minutes after endpoint.

Memory: up to 5 measurements can be stored.

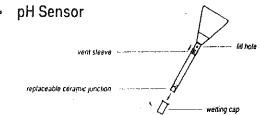
Coming Incorporated Science Products Division Coming, New York 14831 USA Tel: 1-607-737-1667

473655 Rev. A, 3/89

0.1 mg/L

### **Rensor Information**

for direct in...ig and leaded pH, DO and Conductivity Sensors



### For optimum performance:

- 1. Before use remove wetting cap from tip of sensor, and slide the vent sleeve to expose the fill hole.
- 2. Make sure that the fill solution is not more than 25 mm (1 inch) below the till hole. Add KCI solution if necessary.
- Gently tap the sensor to remove any air bubbles at the ceramic junction.
- Condition the new sensor by soaking in pH 7 buffer for 2 hours. Protonged soaking is not recommended.
- 5. Calibrate and measure samples as described in the M90 instructions. Allow sufficient time for the sensor to stabilize when measuring samples of different temperatures, or of low ionic strength. Manual endpointing is advised with these samples.
- After use, check the level of fill solution, reposition the vent sleeve to cover the fill hote, and replace the wetting cap containing pH 7 buffer (if the sensor will not be used again for more than 2 days, we recommend using saturated KCI in the wetting cap).

### Precautions and Limitations:

- Do not wipe the sensor tip blot dry with a lint-free tissue.
- Do not use KCI saturated with AgCI as this may damage the reference
- Do not leave the sensor in organic solvents, strong basic solutions, concentrated fluoride solutions, or hydrofluoric acid for extended periods. Measurements made in these solutions should be taken quickly and the sensor rinsed immediately with distilled water. After rinsing, soak in pH 7 buffer for 2 hours.
- Do not measure solutions that exceed a temperature range of 0 - 100°C.

### Maintenance and Troubleshooting:

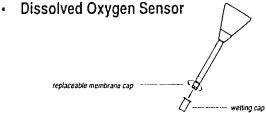
Prolonged use and ageing may reduce performance i.e. slow response, low slope values, continuous drift or erratic readings. These may be caused by: Air in junction - remove air bubbles by gentle tapping.

Excess KCI crystals - KCI crystals may build up and settle on the sensor tip, or the KCI may become discolored. Remove the old fill solution and use warm distilled water to dissolve the crystals. Remove water and refill using fresh KCI solution.

Blocked junction - KCI crystals can block the junction. To test for this, blot the lip dry and air dry for one hour. If no KCI crystals appear at the lip of the sensor the junction is blocked. Remove the ceramic junction using tweezers. and insert new junction (Cat. 477269). Tap gently to remove any air bubbles. Contaminated pH bulb - i.e. protein/oil contamination.

Protein -- soak the sensor in 10% pepsin solution adjusted to pH 2 with HCl

Oil - wash sensor tip with yater-acetone solution. Do not soak the sensor in acetone solutions is may cause the seals to deteriorate. Rinse with distilled water and soak in pH 7 buffer for 2 hours.



### Installation:

DO membrane caps are fragile. Handle with care to prevent damage. The sensor is shipped dry and must be filled before use. Unscrew the membrane cap from the sensor. If the silver/gold tip is tarnished clean carefully using electrode cleaning compound or silver polish, paying particular attention to the gold cathode. Rinse tip with DO electrolyte, and fill membrane cap, avoiding air bubbles. Hold the sensor vertically and gently screw the membrane cap onto the sensor, allowing surplus electrolyte to run out. Fit sensor to the meter and allow 1 hour minimum for polarization. Calibrate as described in M90 instructions.

### For optimum performance:

- Before use remove wetting cap from tip of sensor,
- 2. For immediate use the sensor should be kept connected to the meter. The sensor may be removed for up to 3 hours as a rechargeable battery in the sensor will maintain polarization. For extended storage remove the membrane cap and rinse with water, and clean the sensor tip. Store dry with the membrane cap loosely fitted. Do not fit wetting cap.
- 3. When making measurements the sample should be stirred at a constant speed i.e. approximately 20 cm/second (8 inches/second).
- Allow sufficient time for the sensor to stabilize when measuring samples of different temperatures - in some cases this can be several minutes. Manual endpointing is advised with these samples. Make sure the sensor is immersed to a depth of at least 40 mm (1.5 inches) to cover the temperature sensing element.
- 5. After use replace wetting cap containing distilled water to prevent electrolyte from drying out.
- Regular maintenance is important to ensure optimum performance. Replacement of membrane caps depends on usage - we recommend replacement every 2 to 4 weeks.

### Maintenance and Troubleshooting:

If the sensor will not calibrate, or becomes sluggish or erratic:

- The silver/gold sensor tip may become tarnished with time. For optimum performance clean tip and refill cap every 2 weeks as described in
- 2. The zero oxygen solution will absorb oxygen if left exposed to air and this will cause inaccurate calibration. Use fresh zero oxygen solution.
- Make sure there are no air bubbles inside the membrane cap when filling with DO electrolyte. Check by looking up through the membrane from the bottom of the sensor.
- Check the membrane for damage and replace with new cap (Cat. 473626) as necessary.

Conductivity Sense



### For optimum performance:

- 1. Make sure the clear plastic shield is in place when measurin
- 2. When measuring make sure the solution is above the cell ch and below the vent hole.
- 3. To prevent carryover from high to low conductivity solutions distilled water between measurements.
- Make sure the cell chamber is bubble free when measuring. bubbles, immerse probe in the solution at an angle and then vertical position.
- 5. Allow sufficient time for the sensor to stabilize when measuri different temperatures. Manual endpointing is advised with the
- The sensor is not recommended for low ionic strength solution
- Clean the probe and shield with distilled water after use.

### General Troubleshooting for all Sensors

- To verify meter is working check using the test plug.
- 2. If the sensor connector becomes damaged or wet the display E4 when a sensor is connected.
- 3. If the temperature sensing element becomes damaged the to display may read E1 when a sensor is connected.

### Ordering Information:

pH sensor pH electrode fill solution, 3 x 5 mL pH 7 buffer sachet (pack of 30) pH 4 buffer sachet (pack of 30) pH 10 buffer sachet (pack of 30) Buffer solution pH 4.00, 2 x 500 mL (red) Buffer solution pH 10.01, 2 x 500 mL (blue)

pH multipack, pH 4, 7 and 10 (pack of 30 assorted sachets)

Buffer solution pH 7.00, 2 x 500 mt. (yellow)

Buffer rainbow pack, pH 4.00, 7.00 and 10.01 (2 x 500 mL of each Replaceable ceramic junctions (pH), pack of 3

DO sensor

DO electrolyte, 3 x 5 mL Zero oxygen solution, 500 mL

DO membrane replacement kit, pack of 2

p0, electrode cleaning compound

Conductivity/TDS sensor

1413 µS conductivity standard, 500 mL

12.88 mS conductivity standard, 500 mL

Rinse solution sachet (pack of 30)

Corning Incorporated Science Products Division Corning, New York 14831 Tel: 1-607-737-1667 Technical Information Center: 1-(800)-222-7740

# ATTACHMENT 11G POTENTIOMETRIC MONITORING PLAN AND PROCEDURES

### ATTACHMENT 11G

### Potentiometric Monitoring Plan and Procedures

Although a groundwater monitoring wavier has been granted under R199.9611(b); monitoring of the static water levels at the site will be maintained to verify the continued conditions. If the monitoring determines that hydrogeologic conditions have significantly changed, the director will be notified immediately, as described in Section 11.7.

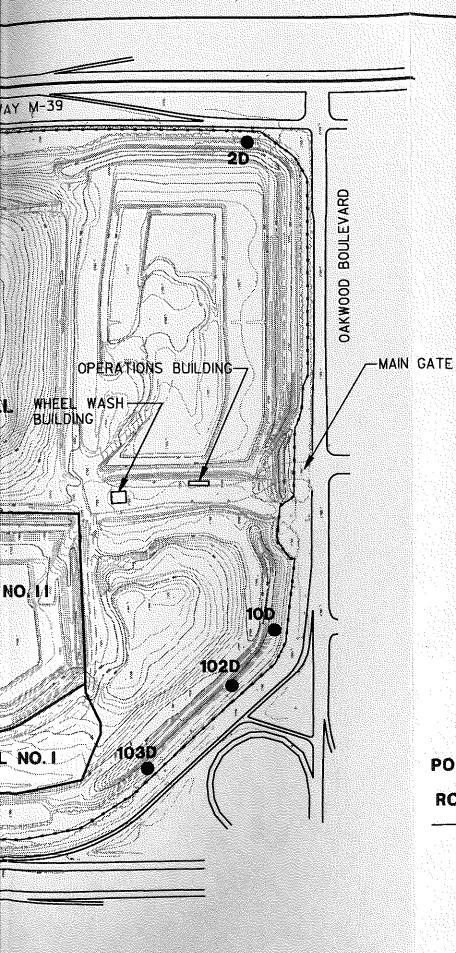
Static water levels have, and will continue to be, measured annually on wells 2D, 5D, 10D, 102D, 103D, 104D, and 105D, which are shown on Figure 11G-1. Except for well 10D, the site monitoring wells are under flowing artesian conditions and are presently outfitted as shown on Figure 11G-2. The well system includes a well plug incorporating a sampling valve. The plug seals the well approximately 2.5 feet below grade level to prevent freezing during winter months. The well plug and valve are constructed of stainless-steel with silicon rubber seals. The valve is used to measure the water level via a staff gauge and a precision pressure gauge. The well may be purged and sampled through this valve as well. Monitoring wells 5D and 103D were constructed using 2-inch, threaded Schedule 40 PVC pipe. These wells have construction configurations similar to that shown on Figure 11G-3.

The procedure for obtaining static water elevations from the outfitted wells are as follows:

- Open the stainless-steel ball valve, allowing groundwater to flow from the system. Once water is observed to discharge from the well, close the ball valve.
- 2. Attach a 4-foot length of Tygon tubing to the closed discharge valve on the well plug system. Attach the other end of the tubing to an inlet valve on a pressure gauge, such as a Wallace and Tiernan Series 1000 Gauge. Calibrate the pressure gauge to read from 0 to 125 inches of water pressure.
- Close the vent valve and then open the discharge valve on the well plug system, allowing water to rise in the Tygon tubing.
- Measure the height of the water in the Tygon tubing (in inches) using a temporary staff gauge. Heights are referenced to the top of the well casing.
- Add the reading of the pressure gauge to the inches of water measured in the Tygon tubing. The total represents the total inches of water above the top of casing elevation.

- 6. Derive the final static water elevation by converting the total inches of water above the casing to feet and adding it to the known USGS casing elevation (recorded in feet) of the well.
- 7. For wells that are not under flowing artesian conditions, measure the depth to water using an electronic water level indicator or a steel tape marked in inches and feet. Record the elevation of the water table by subtracting the depth of water (in feet) from the top of casing elevation referenced to the USGS datum.

All annual water level information will be recorded and filed in chronological order.

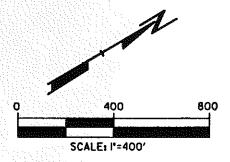


### LEGEND

10D MONITORING WELL

### **NOTES**

- I. BASE MAP TAKEN FROM AN APRIL 1993 AERIAL SURVEY BY ABRAMS AERIAL SURVEY CORP.
- 2. HAZARDOUS WASTE CELL BOUNDARIES ARE BASED ON THE LEGAL DESCRIPTION IN THE RESTRICTIVE COVENANT.



POTENTIOMETRIC MONITORING WELL LOCATIONS RCRA/ACT 64 OPERATING LICENSE <u>APPLICATION</u> FORD MOTOR COMPANY ALLEN PARK, MICHIGAN

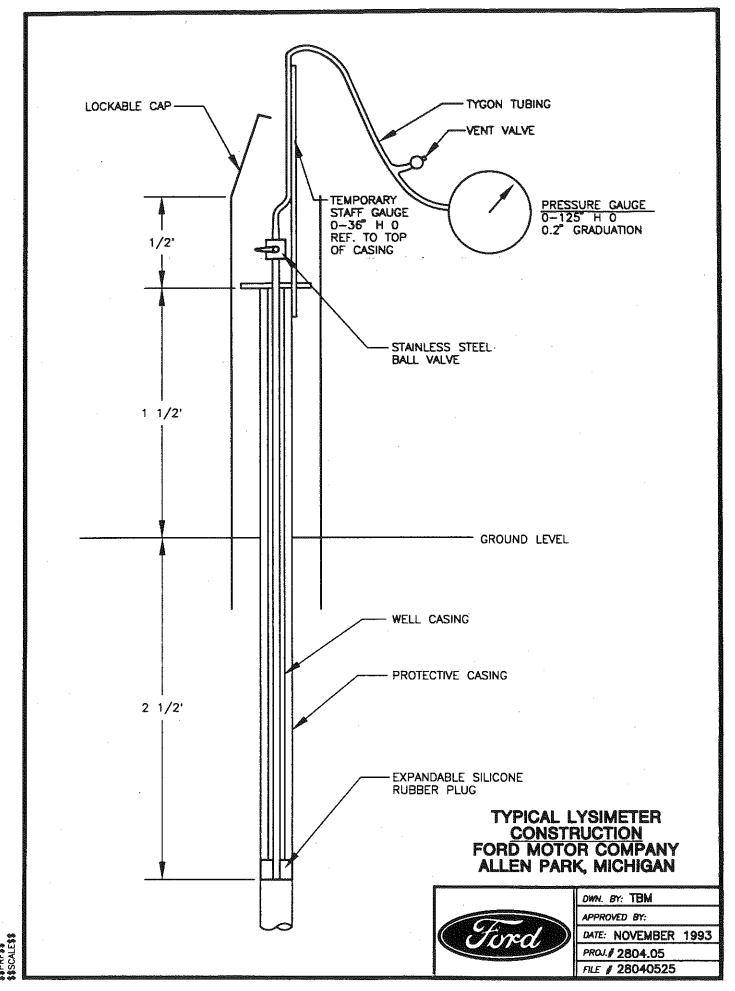


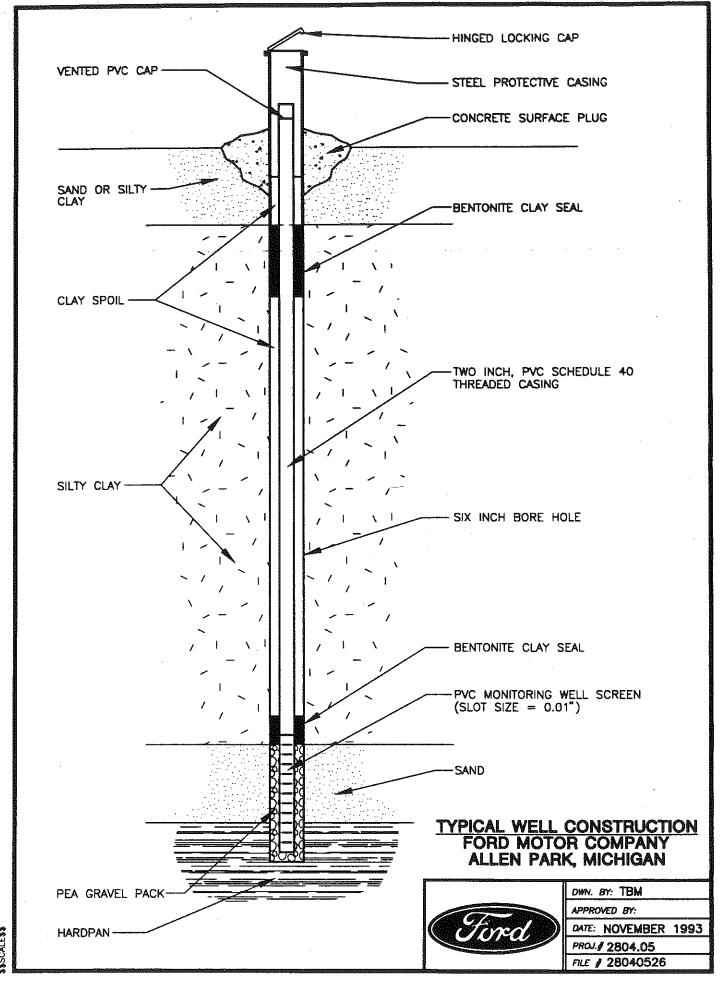
DWN. BY: TBM

APPROVED BY:

PROJ. # 2804.05

FILE # 28040508.dgn





# ATTACHMENT 11H SEDIMENTATION BASIN MONITORING PLAN AND PROCEDURES

### **ATTACHMENT 11H**

### Sedimentation Basin Monitoring Plan and Procedures

Sediment samples will be collected on a semiannual basis after the initiation of filling in Cell II. Sediment sample analytical parameter lists and sample collection procedures are discussed below. Proposed additional background sediment sampling, to be carried out every other month for a period of 1 year, is also discussed below. Procedures for statistical evaluation of sediment data are presented in Subsection 11H.3. A detailed laboratory QA/QC program is provided in Attachment 11A.

### 11H.1 Sediment Sampling Parameters and Schedule

### Additional Background Sediment Sampling

The parameters for the proposed additional background sampling include all of the leachate monitoring parameters listed in Table 11E-1 for which sediment background values have not yet been established. Table 11H-1 identifies all background parameters for which background sediment concentrations have already been established, and which parameters will be sampled for in this additional background sampling. The additional background sampling will consist of collecting duplicate samples at four locations every other month for a period of 1 year. The four sediment sampling locations are identified on Figure 11H-2. Sediment samples will be identified by the station identification numbers used on Figure 11H-2, SD-X1 through SD-X4. The samples will be analyzed only for those parameters for which background values have not yet been established.

### Operational Sediment Sampling Plan

Operational sediment sampling will consist of collecting individual sediment samples on a semiannual basis from each of the four sampling locations. The general sampling area is shown on Figure 11H-1, and sampling locations and identification codes are presented on Figure 11H-2.

The parameter list for operational sediment sampling is presented in Table 11H-2. Sample preservation methods, detection limits, holding times, and analytical methods are presented in Table 11H-3. This list will be reviewed annually and revised if appropriate, as described in Subsection 11.8.2 of this document.

### 11H.2 Sediment Sample Collection

The procedures for sediment sample collection will be as follows:

- Sample collection bottles will be provided pre-preserved from the analytical laboratory. Prepare bottles by labeling according to EPA Chain-of-Custody (COC) requirements using a sample tag as shown on Figure 11H-3, or equivalent. The tag will be filled out in ink and in legible handwriting. In order to maintain consistency with the historical data base, sample identification codes SD-X1 through SD-X4, corresponding to the locations illustrated on Figure 11H-2, will be used.
- Spread a new piece of plastic sheeting on the ground next to each sampling location in order to prevent sampling equipment from contacting the ground. A new pair of disposable PVC or latex gloves will be used by the sampler at each sampling location in order to prevent contamination of the samples. At each selected point, a stainless-steel core sampler will be used to collect a composite sample of materials from the bottom of the sedimentation basin, from a depth of 0 to 6 inches, at a distance of approximately 2 feet from the existing shoreline.
- Fill the sample bottles for volatile organic compounds first. These sample containers must be filled so that no headspace remains in the sample jar.
- 4. Fill remaining sample containers as listed in Table 11H-3. Sample containers will be sealed tightly, and immediately placed on ice.
- 5. All sediment samples will be stored in ice coolers from time of collection through delivery to the analytical laboratory.
- The core sampler will be decontaminated prior to sampling at each location by washing in phosphate-free soapy water followed by double rinse with distilled water.
- 7. Field data sheets will be used to identify the sampling location, time, date, and sampler, along with the sample jars filled at each location. Copies of the field data sheets will be retained at the facility.
- 8. The Chain-of-Custody (COC) Record describing the sample will be drawn up by the sampler. This form will accompany all samples, and be signed and dated by the sampler at the time the samples are released. The individual accepting the samples will also sign and date the same COC form acknowledging receipt of the samples.

### 11H.3 Statistical Evaluation of Sediment Basin Monitoring Data

As soon as possible following each semiannual sampling event, a statistical evaluation of a portion of the data will be conducted to determine if a significant increase of certain parameters has occurred. In order to reduce the facility-wide false positive rate (as discussed in Attachment 11B), statistics will be performed on the following parameters: chrome, copper,

arsenic, selenium, volatile and semi-volatile organic compounds. The list of parameters for which statistical evaluation is performed will be evaluated on an annual basis. Waste codes and leachate analytical data will be reviewed to determine if the statistical evaluation should be expanded to include other parameters which are analyzed for that may provide reliable indicators of environmental impacts at the facility, given changes in waste or leachate composition.

The sediment data will be evaluated on an intra-point basis, so that operational monitoring data from each location will be compared to the background data set from that same sampling location. Follow the procedures outlined below for statistical evaluation. If the reported concentration results in an exceedance of that test statistic, but the reported concentration is less than the appropriate Part 201 of Act 451 Generic Industrial Cleanup Criteria (MDNR Operational Memorandum #14, Revision 2, June 6, 1995) then no action will be taken. If the reported concentration results in an exceedance of the test statistic and the appropriate Part 201 of Act 451 criteria, then the procedure outlined in Subsection 11.8.3 will be followed.

- Tabulate, evaluate, and reduce the existing background data. Review data to determine completeness and to determine if sample locations were consistent and clearly defined. Review any additional background data in the same manner when background data collection is complete.
- 2. Revise, if necessary, the estimated quantitation limits (RDLs) for each constituent. RDLs reported by analytical laboratories may change with time. Use the largest detection limit for each parameter wherever a correction for censored data is needed. Using the largest reported RDL value for each constituent is appropriate because the maximum value sets the level of accuracy that can be attained in future monitoring, even if RDL values decrease in the future.
- 3. If the background data set is 100 percent censored, do not perform statistics. The actual level of detection will serve as the trigger value for resampling. In this case, an operational monitoring sample result which exceeds the analytical detection limit will be confirmed by collecting an individual sample at that location and analyzing for the parameter which exceeded background. This measure is being taken in order to rule out laboratory error as a source of the detection. If the analyte is not detected in the confirmatory sample, no further action will be taken. If the analyte is detected, resample at that location in quadruplicate. Collect the quadruplicate samples within approximately 3 feet of each other so that the spatial variability of concentrations is tested for. If any of the four reported concentrations exceed the detection level, follow the procedures outlined in Subsection 11.8.3.

4. Assess the underlying statistical distribution of the data, and correct for log normality if necessary. After the first round of statistical evaluation has been completed, this step will consist of transforming the current data, if necessary, based on the previous evaluation. As recommended in the February, 1993 USEPA Guidance document, assess normality by constructing probability plots.

Follow the attached guidance for constructing probability plots. The plotted points will approximate a straight line if the data are normal. Construct probability plots of the log-transformed and the raw data for each parameter. Compare the plots and decide which representation of the data is closer to the normal distribution. If the log transform of the data is selected as an appropriate transformation, transform all background and operational data for that sample point for that parameter prior to conducting any statistical tests on the data. State whether the statistical test was conducted on raw or transformed data in all reports regarding the data.

- 5. Inspect the data set for outliers. Conduct formal testing for outliers only if a reported concentration is orders of magnitude higher than the rest of the data set. Follow the procedure for outlier testing presented at the end of this attachment. The outlier test assumes that the data other than the outlier follows a normal distribution. Therefore, if the data set is log-normally distributed (see number 3 above), conduct the outlier test on the log-transformed data. Correct or remove an outlier from the data set only if the value can be identified as:
  - (1) an error in transcription or dilution;
  - (2) a documented error in an analytical procedure or report of matrix interferences in the procedure; or
  - (3) some other factor from those listed in the RCRA guidance (USEPA, 1989; USEPA, 1993).

In the event an outlier can be verified, obtain MDNR permission before removing the outlier from the data set. If no obvious cause can be identified for a value being an outlier, it will remain in the operational data set used for statistical evaluation unless MDNR approval is obtained to remove it.

- 6. Inspect the current round of data for nondetects. If a parameter was reported to be below the RDL for that round, then do not perform a statistical test with that data (i.e., do not perform a statistical evaluation to determine if a nondetect represents an exceedance of background). Add the analytical result to the database for that sample point. This approach is being taken because it is reasonable to assume that a nondetect cannot represent an exceedance of background.
- 7. Evaluate the degree of censorship in the data, and select the appropriate statistical test based on this evaluation. After the first round of statistical evaluation has been completed, this step will consist of evaluating whether the statistical test used during previous rounds remains an appropriate choice for the data set. The following steps will be followed in determining how censored

data will be handled and in choosing the statistical test to be performed for each sampling point:

- a) If the percentage of nondetects in the database for the sample point is less than 15 percent, substitute of a value of 1/2 the RDL for all nondetects and calculate a prediction interval according to the procedure included at the end of this Attachment.
- b) If the percentage of nondetects in the database for the sample point is between 15 and 50 percent, use Cohen's or Aichison's adjustment to calculate the mean and standard deviation of the background data.

  Use these adjusted statistics to calculate a prediction interval. Follow the procedures presented at the end of this attachment to determine which of Cohen's or Aichison's adjustment should be used. Calculate the prediction interval according to the presented procedure.
- c) If the percentage of nondetects in the database for the sample point is between 50 and 90 percent, use the Wilcoxan Rank-Sum Test to compare operational monitoring results to background data. Procedures are attached.
- d) If the percentage of nondetects in the database for the sample point is 90 percent or greater, calculate a Poisson prediction limit. Procedures are included at the end of this Attachment.
- e) If the percentage of nondetects in the background data set is 100 percent follow the procedure listed above in step number 3.
- 8. If the statistical test performed for step number 7 above indicates that an exceedance has occurred, follow the steps outlined in Subsection 11.8.3 of the Permit.

TABLE 11H-1				
BACKGROUND SEDIMENTATION BASIN SAMPLING PARAMETERS				
Parameters for	r which Soil Background Sampling	is Completed:		
Arsenic Barium Cadmium Chromium Copper	Cyanide Lead Mercury Naphthalene Nickel	Phenol Selenium Silver Zinc		
Proposed Ac	Iditional Background Soil Sampling	Parameters:		
Antimony Beryllium Calcium Chloride Cobalt Iron Magnesium	pH Sodium Sulfate Thallium Tin Total recoverable phenolics Vanadium			
Scan 7 F	Polynuclear Aromatic Hydrocarbons	s (PAHs):		
Acenaphthene Acenaphthylene Anthracene Benzo(A)anthracene Benzo(A)pyrene Benzo(B)fluoroanthene	Benzo(G,H,I)perylene Benzo(K)fluoranthene Chrysene Dibenzo(A,H)anthracene Fluoranthene Fluorene	Indeno(1,2,3-CD)pyrene Naphthalene Phenanthrene Pyrene		
V	olatile Organic Compounds (VOCs	)		
Benzene Bromodichloromethane Bromoform Bromomethane Carbon tetrachloride Chlorobenzene Chloroethane 2-Chloroethylvinyl ether Chloroform Chloromethane Dibromochloromethane	1,2-Dichlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene Dichlorodifluoromethane 1,1-Dichloroethane 1,2-Dichloroethane 1,1-Dichloroethene trans-1,2-Dichloroethene 1,2-Dichloropropane cis-1,3-Dichloropropene trans-1,3-Dichloropropene	Ethylbenzene Methylene chloride 1,1,2,2-Tetrachloroethane Tetrachloroethene Toluene 1,1,1-Trichloroethane 1,1,2-Trichloroethane Trichloroethene Trichloroethene Trichlorofluoromethane Vinyl chloride		
NOTES:				

### NOTES:

 VOCs include purgeable halocarbons and aromatics listed in Appendix A to Part 136 (40 CFR) - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater.

TABLE 11H-2			
SEDIMENTATION BASIN OPERATIONAL MONITORING PARAMETERS			
	Total Metals		
Antimony Arsenic Barium Beryllium Cadmium Chromium	Cobalt Copper Lead Mercury Nickel Selenium	Silver Thallium Tin Vanadium Zinc	
	Inorganics and Other Parameters		
Calcium Chloride Cyanide	Iron Magnesium pH	Sodium Sulfate Total recoverable phenolics	
Scar	n 7 Polynuclear Aromatic Hydrocar	bons	
Acenaphthene Acenaphthylene Anthracene Benzo(A)anthracene Benzo(A)pyrene Benzo(B)fluoroanthene	Benzo(G,H,I)perylene Benzo(K)fluoranthene Chrysene Dibenzo(A,H)anthracene Fluoranthene Fluorene	Indeno(1,2,3-CD)pyrene Naphthalene Phenanthrene Pyrene	
٧	olatile Organic Compounds (VOCs	r)	
Benzene Bromodichloromethane Bromoform Bromomethane Carbon tetrachloride Chlorobenzene Chloroethane 2-Chloroethylvinyl ether Chloroform Chloromethane Dibromochloromethane	1,2-Dichlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene Dichlorodifluoromethane 1,1-Dichloroethane 1,2-Dichloroethene trans-1,2-Dichloroethene 1,2-Dichloropropane cis-1,3-Dichloropropene trans-1,3-Dichloropropene	Ethylbenzene Methylene chloride 1,1,2,2-Tetrachloroethane Tetrachloroethene Toluene 1,1,1-Trichloroethane 1,1,2-Trichloroethane Trichloroethene Trichlorofluoromethane Vinyl chloride	

### NOTES:

- VOCs include purgeable halocarbons and aromatics listed in Appendix A to Part 136 (40 CFR) Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater. This list will be reviewed annually and revised if appropriate as discussed in 1.
- 2. Subsection 11.8.2.

TABLE 11H-3
SEDIMENT SAMPLING PARAMETERS AND METHODS

Parameter	Analytical Method <sup>1</sup>	Estimated Detection Limit	Container & Preservative	Holding Time
Antimony	6010	2.5 mg/kg	G	6 months
Arsenic	7061	0.5 mg/kg	G	6 months
Barium	6010	1 mg/kg	Ġ	6 months
Beryllium	·6020	-0:2 mg/kg	~ <b>G</b>	6 months
Cadmium	6010	0.5 mg/kg	G	6 months
Chromium	6010	1.0 mg/kg	G	6 months
Cobalt	6010	5.0 mg/kg	G	6 months
Copper	6010	1 mg/kg	G	6 months
Lead	6010	5.0 mg/kg	G	6 months
Mercury	7471	0.1 mg/kg	G	38 days
Nickel	6010	5 mg/kg	G	38 days
Selenium	7741	0.5 mg/kg	G	6 months
Silver	6020	0.25 mg/kg	G	6 months
Thallium	6010	1.0 mg/kg	G	6 months
Tin	6010	5.0 mg/kg	G	6 months
Vanadium	6010	1.0 mg/kg	G	6 months
Zinc	6010	1 mg/kg	G	6 months
lron	6010	2.5 mg/kg	G	6 months
Bicarbonate alkalinity	310.1*	200 mg/kg	G	None specified
Carbonate alkalinity	310.1*	200 mg/kg	G	None specified
Calcium	6010	50 mg/kg	G	6 months
Cyanide	4500	2.5 mg/kg	Р	14 days
Magnesium	6010	50 mg/kg	G	6 months
Sodium	6010	50 mg/kg	G	6 months
Chloride	325.2*	20 mg/kg	G	None specified
Sulfate	375.4*	40 mg/kg	G	None specified
Total recoverable phenolics	9065	0.2 mg/kg	G	None specified
Benzene	8260	5.0 mg/kg	G, T, A	14 days
Ethylbenzene	8260	5.0 mg/kg	G, T, R	14 days
Toluene	8260	5.0 mg/kg	G, T, R	14 days

TABLE 11H-3
SEDIMENT SAMPLING PARAMETERS AND METHODS

Parameter	Analytical Method <sup>1</sup>	Estimated Detection Limit	Container & Preservative	Holding Time
Vinyl chloride	8260	10.0 mg/kg	G, T, R	14 days
Bromodichloromethane	8260	5.0 mg/kg	G, T, A	14 days
Bromoform	8260	5.0 mg/kg	G, T, R	14 days
Bromomethane	8260	10.0-mg/kg	.G, <b>Т,</b> -R	- 14 days
Carbon tetrachloride	8260	5.0 mg/kg	G, T, R	14 days
Chlorobenzene	8260	5.0 mg/kg	G, T, R	14 days
Chloroethane	8260	10.0 mg/kg	G, T, R	14 days
2-Chloroethylvinyl ether	8260	5.0 mg/kg	G, T, R	14 days
Chloroform	8260	5.0 mg/kg	G, T, R	14 days
Chloromethane	8260	10.0 mg/kg	G, T, R	14 days
Dibromochloromethane	8260	5.0 mg/kg	G, T, R	14 days
1,2-Dichlorobenzene	8260	5.0 mg/kg	G, T, R	14 days
1,3-Dichlorobenzene	8260	5.0 mg/kg	G, T, R	14 days
1,4-Dichlorobenzene	8260	5.0 mg/kg	G, T, R	14 days
Dichlorodifluoromethane	8260	10.0 mg/kg	G, T, R	14 days
1,1-Dichloroethane	8260	5.0 mg/kg	G, T, R	14 days
1,2-Dichloroethane	8260	5.0 mg/kg	G, T, R	14 days
1,1-Dichloroethene	8260	5.0 mg/kg	G, T, R	14 days
trans-1,2-Dichloroethene	8260	5.0 mg/kg	G, T, R	14 days
1,2-Dichloropropane	8260	5.0 mg/kg	G, T, A	14 days
cis-1,3-Dichloropropene	8260	5.0 mg/kg	G, T, R	14 days
trans-1,3-Dichloropropene	8260	5.0 mg/kg	G, T, R	14 days
Methylene chloride	8260	10.0 mg/kg	G, T, R	14 days
1,1,2,2-Tetrachloroethene	8260	5.0 mg/kg	G, T, R	14 days
Tetrachloroethene	8260	5.0 mg/kg	G, T, R	14 days
1,1,1-Tr <del>i</del> chloroethane	8260	5.0 mg/kg	G, T, R	14 days
1,1,2-Trichloroethane	8260	5.0 mg/kg	G, T, R	14 days
Trichlorofluoromethene	8260	10.0 mg/kg	G, T, R	14 days
Acenaphthene	8270	200 mg/kg	G, R	F
Acenaphthylene	8270	200 mg/kg	G, R	F

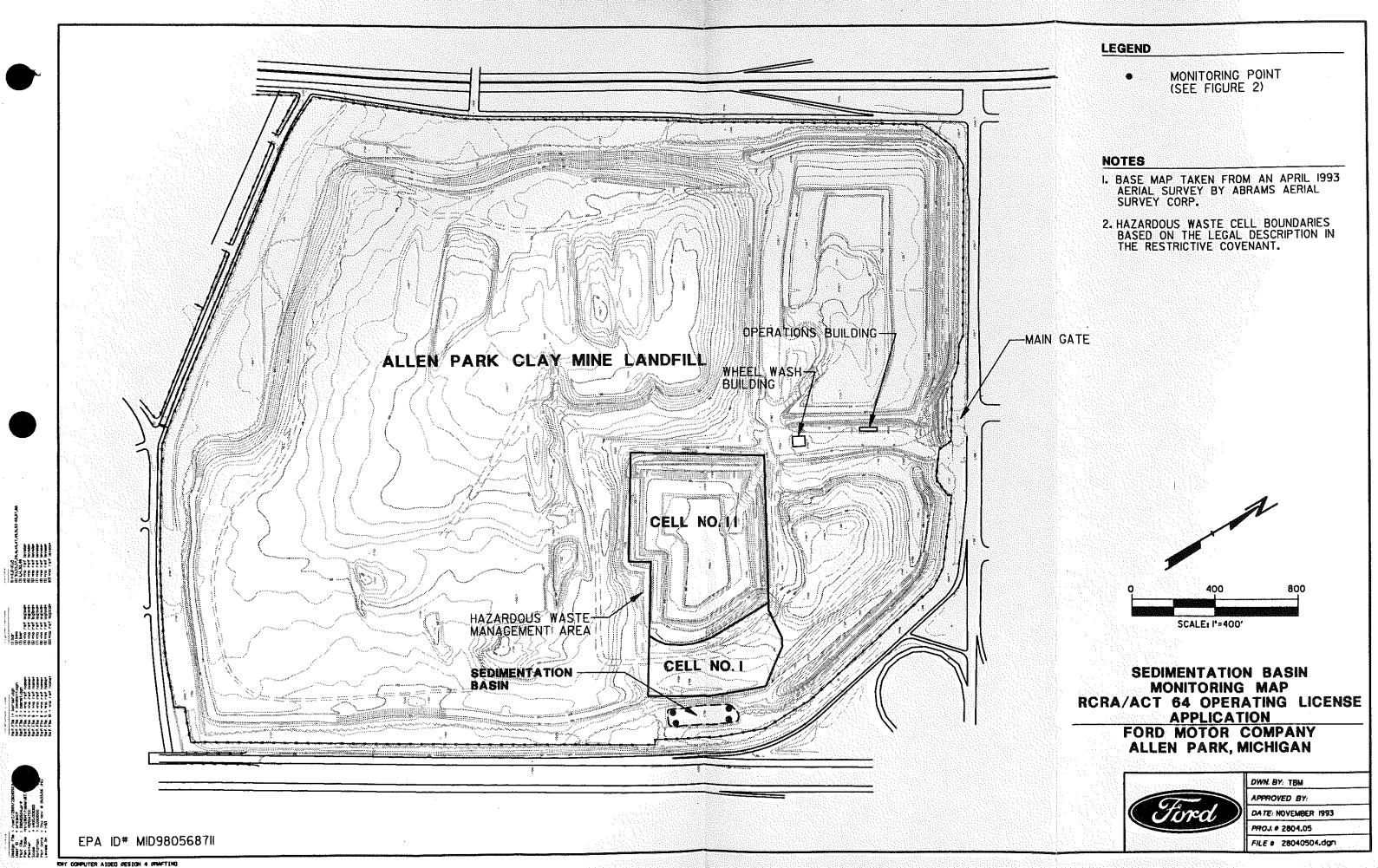
### TABLE 11H-3

### **SEDIMENT SAMPLING PARAMETERS AND METHODS**

Parameter	Analytical Method <sup>1</sup>	Estimated Detection Limit	Container & Preservative	Holding Time
Anthracene	8270	250 mg/kg	G, R	F
Benzo(A) anthracene	8270	200 mg/kg	G, R	F
Benzo(A) pyrene	8270	200 mg/kg	G, R	F
Benzo(B)fluoranthene	8270	200 mg/kg	G, R	F
Benzo(G,H,I)fluoranthene	8270	330 mg/kg	G, R	F
Benzo(K)fluoranthene	8270	250 mg/kg	and the second	
Chrysene	8270	200 mg/kg	G, R	F
Dibenzo(A,H) anthracene	8270	330 mg/kg	G, R	F
Fluoranthene	8270	300 mg/kg	G, R	F
Fluorene	8270	200 mg/kg	G, R	F
Indeno(1,2,3-CD) pyrene	8270	330 mg/kg	G, A	F
Naphthalene	8270	200 mg/kg	G, R	F
Phenanthrene	8270	200 mg/kg	G, R	F
Pyrene	8270	200 mg/kg	G, R	F

### NOTES:

- "Test Methods for Evaluating Solid Waste," SW-846, Third Edition.
- 2 USEPA Methods 600/4-79/200.
- Requires performance of ASTM Leach (D3987-85) prior to wet chemistry analysis.
- Р Plastic.
- G Glass.
- Refrigeration. R
- Nitric acid to pH <2. Ν
- Teflon®-lined cap.
- 7 days to extraction and 40 days from extraction. Sulfuric acid to pH <2. Е
- S
- AG Amber glass.
- Hydrochloric acid to pH <2.



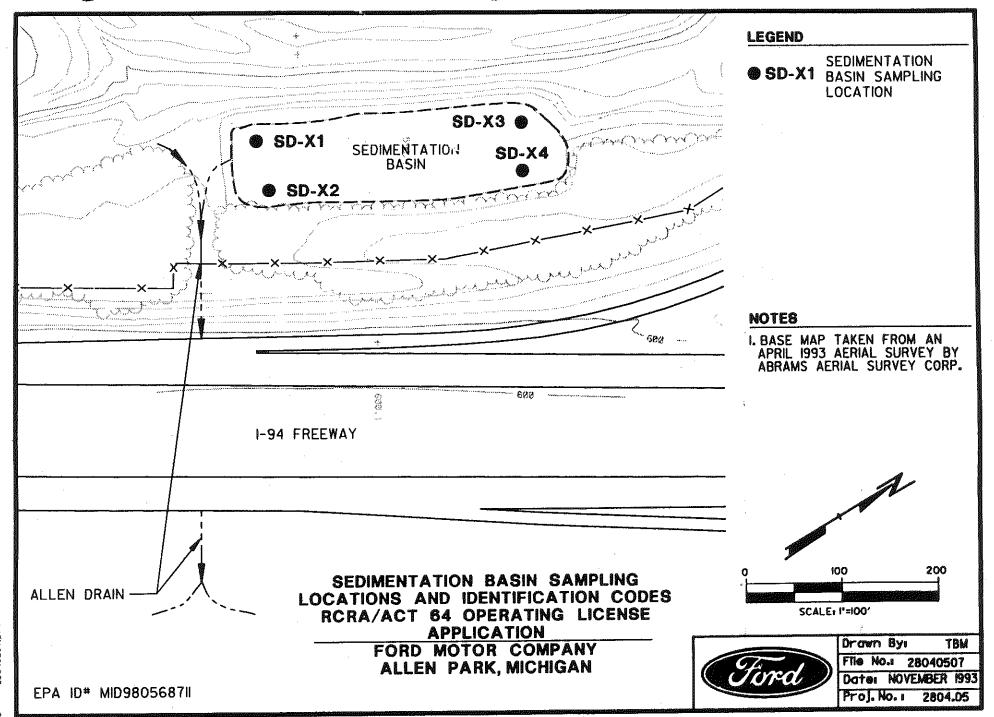


FIGURE 11H-2

	· ·		Ford Allen Park Clay Mine Ford Motor Company Sediment Pond Sediment
	Date		
	Time		
	Location		
((<))	Analyze for		
	Preserved with	- Carrier Control of the Control of	and the state of t
// // . \	Samplers Signatures		
<b>/</b> //	Time	Date	

EACH SAMPLE TRANSFERRED SHOULD HAVE A LABEL ATTACHED.

SAMPLE LABEL
FORD MOTOR COMPANY
ALLEN PARK, MICHIGAN



OWN. BY: TBM

APPROVED BY:

DATE: NOVEMBER 1993

PROJ.# 2804.05

FILE # 28040515

### ATTACHMENT 11I LEAK DETECTION MONITORING PLAN AND PROCEDURES

#### **ATTACHMENT 11**

### Leak Detection Monitoring Plan and Procedures

Water samples from the leak detection monitoring system of Cell II will be collected on an annual basis after the initiation of filling in Cell II, if sufficient water is present in the monitoring sump. Leak detection analytical parameter lists and sample collection procedures are discussed below. Proposed additional background leak detection sampling, to be carried out every other month for a period of 1 year, is also discussed below. Statistical procedures for evaluating data are presented in Subsection 11D.3. A detailed laboratory QA/QC program is provided in Attachment 11A.

### 111.1 Leak Detection Sampling Parameters and Schedule

### Additional Background Leak Detection System Sampling

The parameters for the proposed additional background sampling include all of the leachate monitoring parameters for which leak detection background values have not already been established. Table 11I-1 identifies those parameters for which background leak detection sampling is completed for, and which parameters are proposed for additional background sampling. The additional background sampling will consist of sampling every other month for a period of 1 year. Each sampling event will consist of collecting an individual sample at the monitoring sump of the leak detection system for Cell II, if a sufficient volume of water is present. Sample preservation methods, detection limits, holding times, and analytical methods are presented in Table 11I-3. Background leak detection samples will be identified by the station i.d. associated with the monitoring sump. Field and sample handling procedures are discussed below.

### Operational Leak Detection System Sampling Plan

Operational leak detection sampling will consist of collecting an individual water sample at the sump on a quarterly basis, if sufficient water is present. The parameter list for operational monitoring of the leak detection system is presented in Table 11I-2. Sample volumes, preservation methods, detection limits, holding times, and analytical methods are presented in Table 11I-3. The operational monitoring parameter list (Table 11I-2) will be reviewed annually and revised if appropriate, as described in Subsection 11.3.2 of this application and in Attachment 11E, the Leachate Sampling and Monitoring Plan. In addition to quarterly samples for chemical analysis, the volumes of water collected in the sump and discharged to the

sanitary sewer system will be recorded on a monthly basis. The records of volumes will be kept on file at the facility office.

#### 111.2 Leak Detection Sample Collection

Providing that sufficient volumes can be obtained, samples for chemical analysis will be collected from the monitoring sumps according to the following procedure:

- 1. Sample collection bottles will be provided pre-preserved from the analytical laboratory. Prepare bottles by labeling according to EPA Chain-of-Custody (COC) requirements using a sample tag as shown on Figure 11F-3, or equivalent. The tag will be filled out in ink and in legible handwriting.
- 2. Spread a new piece of plastic sheeting on the ground next to the leak detection sump sampling location in order to prevent sampling equipment from contacting the ground. A new pair of disposable PVC or latex gloves will be used by the sampler at each sampling location in order to prevent contamination of the samples.
- Collect samples using a dedicated pump (EPG TSP1-3 or equivalent).
- 4. Sample vials for volatile organics should be filled first. The sample should be collected in a manner which minimizes sample disturbance. The sample stream should be allowed to strike the inner wall of the vial to minimize formation of air bubbles. Fill the sample vial or bottle with a minimum of splashing. Fill each vial until the water forms a positive meniscus at the brim. Allow the vial to overflow slightly, then replace the cap by gently setting it on the water meniscus. Tighten firmly, but do not over-tighten.
- 5. Invert the vial and tap lightly to check for air bubbles.
- 6. Place samples on ice immediately.
- 7. Fill appropriate bottles for other sample parameters. Note that leak detection system samples are not field filtered for any analytical parameters. Place samples in coolers with ice immediately following sample collection. All samples will be stored in ice coolers from time of collection through delivery to the analytical laboratory.
- 8. Fill a disposable plastic sample cup with 200 to 300 mL of water from the leak detection monitoring point and collect temperature, pH and specific conductance measurements. These measurements will be collected with a Corning Model M90 pH and conductivity meter, or equivalent, according to the manufacturer's specifications. The pH and conductivity probes will be cleaned between sampling locations by double rinsing with distilled water. The Model M90 meter will be calibrated once prior to use for every 4 hours of use in the field, according to the manufacturer's specifications for calibration. Manufacturer's specifications for instrument use and calibration are included at the end of this Attachment.

- 9. Field measurements will be recorded on field data sheets, which will identify the sampling location, time, date, and sampler, along with the measurements collected and a description of the number ant type of sample bottles filled. Copies of the field data sheets will be retained at the facility.
- 10. The Chain-of-Custody (COC) Form describing the sample will be drawn up by the sampler. This form will accompany all samples, and be signed and dated by the sampler at the time the samples are released. The individual accepting the samples will also sign and date the same COC form acknowledging receipt of the samples. Copies of the COC will be retained at the facility.

# 111.3 Statistical Evaluation of Leak Detection System Monitoring Data

As soon as possible following each quarterly sampling event, a statistical evaluation of a portion of the data will be conducted to determine if a significant increase of certain parameters has occurred. In order to reduce the facility-wide false positive rate (as discussed in Attachment 11B), statistics will be performed on the following parameters: chrome, copper, arsenic, selenium, volatile and semi-volatile organic compounds. The list of parameters for which statistical evaluation is performed will be evaluated on an annual basis. Waste codes and leachate analytical data will be reviewed to determine if the statistical evaluation should be expanded to include other parameters which are analyzed for that may provide reliable indicators of environmental impacts at the facility, given changes in waste or leachate composition.

The leak detection system data will be evaluated on an intra-point basis, so that operational monitoring data from the leak detection sump will be compared to the background data set from that same sampling location. Follow the procedures outlined below for statistical evaluation. If the reported concentration results in an exceedance of that test statistic, then the procedure outlined in Subsection 11.9.3 will be followed.

- Tabulate, evaluate, and reduce the existing background data. Review data to determine completeness and to determine if sample locations were consistent and clearly defined. Review any additional background data in the same manner when background data collection is complete.
- 2. Revise, if necessary, the estimated quantitation limits (RDLs) for each constituent. RDLs reported by analytical laboratories may change with time. Use the largest detection limit for each parameter wherever a correction for censored data is needed. Using the largest reported RDL value for each constituent is appropriate because the maximum value sets the level of accuracy that can be attained in future monitoring, even if RDL values decrease in the future.

- 3. If the background data set is 100 percent censored, do not perform statistics. The actual level of detection will serve as the trigger value for resampling. In this case, an operational monitoring sample result which exceeds the analytical detection limit will be confirmed by collecting an individual sample at that location and analyzing for the parameter which exceeded background. This measure is being taken in order to rule out laboratory error as a source of the detection. If the analyte is not detected in the confirmatory sample, no further action will be taken. If the analyte is detected, resample at that location in quadruplicate. Evaluate the resampling data by following step number 6 below, using the statistical procedure appropriate for the percentage of nondetects in the results.
- -4. Assess the underlying statistical distribution of the data, and correct for log normality if necessary. After the first round of statistical evaluation has been completed, this step will consist of transforming the current data, if necessary, based on the previous evaluation. As recommended in the February, 1993 USEPA Guidance document, assess normality by constructing probability plots.

Follow the attached guidance for constructing probability plots. The plotted points will approximate a straight line if the data are normal. Construct probability plots of the log-transformed and the raw data for each parameter. Compare the plots and decide which representation of the data is closer to the normal distribution. If the log transform of the data is selected as an appropriate transformation, transform all background and operational data for that sample point for that parameter prior to conducting any statistical tests on the data. State whether the statistical test was conducted on raw or transformed data in all reports regarding the data.

- 5. Inspect the data set for outliers. Conduct formal testing for outliers only if a reported concentration is orders of magnitude higher than the rest of the data set. Follow the procedure for outlier testing presented at the end of this attachment. The outlier test assumes that the data other than the outlier follows a normal distribution. Therefore, if the data set is log-normally distributed (see number 3 above), conduct the outlier test on the log-transformed data. Correct or remove an outlier from the data set only if the value can be identified as:
  - (1) an error in transcription or dilution;
  - (2) a documented error in an analytical procedure or report of matrix interferences in the procedure; or
  - (3) some other factor from those listed in the RCRA guidance (USEPA, 1989; USEPA, 1993).

In the event an outlier can be verified, obtain MDNR permission before removing the outlier from the data set. If no obvious cause can be identified for a value being an outlier, it will remain in the operational data set used for statistical evaluation unless MDNR approval is obtained to remove it.

- 6. Inspect the current round of data for nondetects. If a parameter was reported to be below the RDL for that round, then do not perform a statistical test with that data (i.e., do not perform a statistical evaluation to determine if a nondetect represents an exceedance of background). Add the analytical result to the database for that sample point. This approach is being taken because it is reasonable to assume that a nondetect cannot represent an exceedance of background.
- 7. Evaluate the degree of censorship in the data, and select the appropriate statistical test based on this evaluation. After the first round of statistical evaluation has been completed, this step will consist of evaluating whether the statistical test used during previous rounds remains an appropriate choice for the data set. The following steps will be followed in determining how censored data will be handled and in choosing the statistical test to be performed for each sampling point:
  - a) If the percentage of nondetects in the database for the sample point is less than 15 percent, substitute of a value of 1/2 the RDL for all nondetects and calculate a prediction interval according to the procedure included at the end of this Attachment.
  - b) If the percentage of nondetects in the database for the sample point is between 15 and 50 percent, use Cohen's or Aichison's adjustment to calculate the mean and standard deviation of the background data. Use these adjusted statistics to calculate a prediction interval. Follow the procedures presented at the end of this attachment to determine which of Cohen's or Aichison's adjustment should be used. Calculate the prediction interval according to the presented procedure.
  - c) If the percentage of nondetects in the database for the sample point is between 50 and 90 percent, use the Wilcoxan Rank-Sum Test to compare operational monitoring results to background data. Procedures are attached.
  - d) If the percentage of nondetects in the database for the sample point is 90 percent or greater, calculate a Poisson prediction limit. Procedures are included at the end of this Attachment.
  - e) If the percentage of nondetects in the background data set is 100 percent follow the procedure listed above in step number 3.
- 8. If the statistical test performed for step number 7 above indicates that an exceedance has occurred, follow the steps outlined in Subsection 11.9 of the Permit.

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# NOTES:

- 1.
- Leak detection samples are analyzed for total metals and are therefore not field-filtered. VOCs include purgeable halocarbons and aromatics listed in Appendix A to Part 136 2. (40 CFR) - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater.

TABLE 11I-2					
LEAK DETECTION OPERATIONAL MONITORING PARAMETERS					
	Total Metals				
Antimony Arsenic Barium Beryllium Cadmium Chromium	Cobalt Copper Lead Mercury Nickel Selenium	Silver Thallium Tin Vanadium Zinc			
	Inorganics and Other Parameters	A STATE OF THE STA			
Bicarbonate alkalinity Carbonate alkalinity Calcium Chloride	Cyanide Iron Magnesium pH*	Sodium Specific Conductance* Sulfate Total recoverable phenolics			
Scar	7 Polynuclear Aromatic Hydrocar	bons			
Acenaphthene Acenaphthylene Anthracene Benzo(A)anthracene Benzo(A)pyrene Benzo(B)fluoroanthene	Benzo(G,H,I)perylene Benzo(K)fluoranthene Chrysene Dibenzo(A,H)anthracene Fluoranthene Fluorene	Indeno(1,2,3-CD)pyrene Naphthalene Phenanthrene Pyrene			
V	olatile Organic Compounds (VOCs	)			
Benzene Bromodichloromethane Bromoform Bromomethane Carbon tetrachloride Chlorobenzene Chloroethane 2-Chloroethylvinyl ether Chloroform Chloromethane Dibromochloromethane	1,2-Dichlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene Dichlorodifluoromethane 1,1-Dichloroethane 1,2-Dichloroethene trans-1,2-Dichloroethene 1,2-Dichloropropane cis-1,3-Dichloropropene trans-1,3-Dichloropropene	Ethylbenzene Methylene chloride 1,1,2,2-Tetrachloroethane Tetrachloroethene Toluene 1,1,1-Trichloroethane 1,1,2-Trichloroethane Trichloroethene Trichloroethene Trichlorofluoromethane Vinyl chloride			

# NOTES:

- 1. Leak detection samples are analyzed for total metals and are therefore not field-filtered.
- VOCs include purgeable halocarbons and aromatics listed in Appendix A to Part 136 (40 CFR) - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater.
- 3. This list will be reviewed annually and revised if appropriate as discussed in Subsection 11.9.2.

TABLE 11I-3

LEAK DETECTION SAMPLING PARAMETERS AND METHODS

Parameter	Analytical Method <sup>1</sup>	Estimated Detection Limit	Container & Preservative	Holding Time
Antimony (total)	6020	1 μg/L	G, N	6 months
Arsenic (total)	6020, 7061	1 #g/L	G, N	6 months
Barium (total)	6020	5 μg/L	G, N	6 months
Beryllium (total)	~ 6020	1-#g/L ···	-G,-N ·	6 months
Cadmium (total)	6020	0.2 μg/L	G, N	6 months
Chromium (total)	6020	1 μg/L	G, N	6 months
Cobalt (total)	6010	15 μg/L	G, N	6 months
Copper (total)	6010	1 μg/L	G, N	6 months
Lead (total)	6020	1.0 μg/L	G, N	6 months
Mercury (total)	7470	0.2 μg/L	G, N	38 days
Nickel (total)	6020	50 μg/L	G, N	6 months
Selenium (total)	6020, 7741	1 μg/L	G, N	6 months
Silver (total)	6020	0.5 µg/L	G, N	6 months
Thallium (total)	6020	2.0 μg/L	G, N	6 months
Tin (total)	200.7 <sup>2</sup>	500 μg/L	G, N	6 months
Vanadium (total)	6010	10 µg/L	G, N	6 months
Zinc (total)	6020	4 μg/L	G, N	6 months
iron (total)	6010	0.02 mg/L	G, N	6 months
Bicarbonate alkalinity	310.1	5 mg/L	P, R	14 days
Carbonate alkalinity	310.1	10 mg/L	P, R	14 days
Calcium	6010	1 mg/L	G, N	6 months
Cyanide	335.2	5 μg/L	P, H	14 days
Magnesium	6010	1 mg/L	G, N	6 months
Sodium	6010	1 mg/L	G, N	6 months
Chloride	35.2	1 mg/L	P, R	28 days
Sulfate .	375.4	2 mg/L	P, R	28 days
Total recoverable phenolics	9065	0.01 mg/L	AG, T, S	28 days
Benzene	8260	1.0 mg/L	C, G, T, R	14 days
Ethylbenzene	8260	1.0 mg/L	C, G, T, R	14 days
Toluene	8260	1.0 mg/L	C, G, T, R	14 days

TABLE 11I-3

LEAK DETECTION SAMPLING PARAMETERS AND METHODS

Parameter	Analytical Method <sup>1</sup>	Estimated Detection Limit	Container & Preservative	Holding Time
Vinyl chloride	8260	5.0 mg/L	C, G, T, R	14 days
Bromodichloromethane	8260	1.0 mg/L	C, G, T, R	14 days
Bromoform	8260	1.0 mg/L	C, G, T, R	14 days
Bromomethane	8260	5.0 mg/L	С, G, Л, Я	-14 days
Carbon tetrachloride	8260	1.0 mg/L	C, G, T, R	14 days
Chlorobenzene	8260	1.0 mg/L	C, G, T, R	14 days
Chloroethane	8260	5.0 mg/L	C, G, T, R	14 days
2-Chloroethylvinyl ether	8260	5.0 mg/L	C, G, T, R	14 days
Chloroform	8260	1.0 mg/L	C, G, T, R	14 days
Chloromethane	8260	5.0 mg/L	C, G, T, R	14 days
Dibromochloromethane	8260	1.0 mg/L	C, G, T, R	14 days
1,2-Dichlorobenzene	8260	1.0 mg/L	C, G, T, R	14 days
1,3-Dichlorobenzene	8260	1.0 mg/L	C, G, T, R	14 days
1,4-Dichlorobenzene	8260	1.0 mg/L <sup></sup>	C, G, T, R	14 days
Dichlorodifluoromethane	8260	5.0 mg/L	C, G, T, R	14 days
1,1-Dichloroethane	8260	1.0 mg/L	C, G, T, R	14 days
1,2-Dichloroethane	8260	1.0 mg/L	C, G, T, R	14 days
1,1-Dichloroethene	8260	1.0 mg/L	C, G, T, R	14 days
trans-1,2-Dichloroethene	8260	1.0 mg/L	C, G, T, R	14 days
1,2-Dichloropropane	8260	1,0 mg/L	C, G, T, R	14 days
cis-1,3-Dichloropropene	8260	1.0 mg/L	C, G, T, R	14 days
trans-1,3-Dichloropropene	8260	1.0 mg/L	C, G, T, R	14 days
Methylene chloride	8260	5.0 mg/L	C, G, T, R	14 days
1,1,2,2-Tetrachloroethene	8260	1.0 mg/L	C, G, T, R	14 days
Tetrachioroethene	8260	1.0 mg/L	C, G, T, R	14 days
1,1,1-Trichloroethane	8260	1.0 mg/L	C, G, T, R	14 days
1,1,2-Trichloroethane	8260	1.0 mg/L	C, G, T, R	14 days
Trichloroethene	8260	1.0 mg/L	C, G, T, R	14 days

TABLE 11I-3

LEAK DETECTION SAMPLING PARAMETERS AND METHODS

Parameter	Analytical Method <sup>1</sup>	Estimated Detection Limit	Container & Preservative	Holding Time
2,4,5-Trichlorophenol	8270	10 µg/L	AG, R	E
2,6,4-Trichlorophenol	8270	10 µg/L	AG, R	E
2,4-Dichlorophenol	8270	10 μg/L	AG, P	E
2,4-Dimethylphanol	8270	10-µg/L	-AG,-R	, €
2,4-Dinitrophenol	8270	50 μg/L	AG, R	E
2-Chlorophenol	8270	10 µg/L	AG, R	E
2-Nitrophenol	8270	10 µg/L	AG, R	E
4-Nitrophenol	8270	50 μg/L	AG, R	E
2-Methyl-4,6-dinitrophenol	8270	50 μg/L	AG, A	Ε
Pentachlorophenol	8270	50 μg/L	AG, R	E
4-Chloro-3-methylphenol	8270	10 μg/L	AG, R	E
Phenol	8270	10 µg/L	AG, R	Ε
Acenaphthene	8310	1.0 μg/L	AG, R	E
Acenaphthylene	8310	1.0 μg/L	AG, R	E
Anthracene	8310	1.0 μg/L	AG, R	E
Benzo(A) anthracene	8310	1.0 μg/L	AG, R	E
Benzo(A) pyrene	8310	2.0 μg/L	AG, R	E
Benzo(B)fluoranthene	8310	2.0 μg/L	AG, R	E
Benzo(G,H,I)fluoranthene	8310	5.0 μg/L	AG, R	E
Benzo(K)fluoranthene	8310	2.0 μg/L	AG, R	E
Chrysene	8310	1.0 μg/L	ÁG, R	E
Dibenzo(A,H) anthracene	8310	5.0 μg/L	AG, R	E
Fluoranthene	8310	1.0 μg/L	AG, R	Ε
Fluorene	8310	1.0 μg/L	AG, R	E

# **TABLE 111-3**

# LEAK DETECTION SAMPLING PARAMETERS AND METHODS

Parameter	Analytical Method¹	Estimated Detection Limit	Container & Preservative	Holding Time
Indeno(1,2,3-CD)pyrene	8310	5.0 μg/L	AG, R	E
Naphthalene	8310	1.0 gg/L	AG, R	E
Phenanthrene	8310	1.0 gg/L	AG, R	E
Pyrene	8310	1.0.µg/L	₋AG, R	E

# NOTES:

- "Test Methods for Evaluating Solid Waste," SW-846, Third Edition.
- USEPA Methods 600/4-79/200.
- Plastic.
- G Glass.
- Refrigeration.
- N Nitric acid to pH <2.
- T Teflon®-lined cap.
- 7 days to extraction and 40 days from extraction.
- S Sulfuric acid to pH <2.
- AG Amber glass.
- Hydrochloric acid to pH <2. NaOH to pH >12. C

		Ford Allen Park Clay Mir Ford Motor Company Leak Detection System	18
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EACH SAMPLE TRANSFERRED SHOULD HAVE A LABEL ATTACHED.

SAMPLE LABEL
FORD MOTOR COMPANY
ALLEN PARK, MICHIGAN



DWN. BY: TBM APPROVED BY:

DATE: NOVEMBER 1993

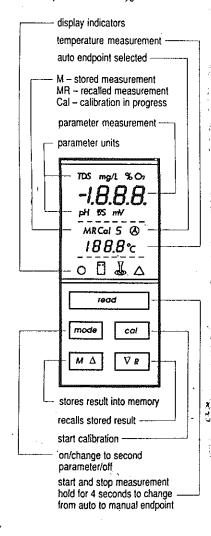
PROJ.# 2804.05 FILE # 28040519 **EQUIPMENT OPERATION AND CALIBRATION MANUALS** 

# **M90 Operating Instructions**

The M90 is a portable, microprocessor based, pH, conductivity and dissolved oxygen meter.

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# Installing the Battery . . .

Remove rubber battery door on the rear of the meter. Fit battery — alkaline type 9V PP3/1604 or equivalent. Make sure polarity is correct when fitting. The meter must be calibrated after replacing the battery.

#### Installing a Sensor . . .

Locate the pins of the sensor in the meter and push firmly into the meter. The sensor is removed by squeezing down the catch at the rear and pulling the sensor away from the meter.

#### Making a Measurement . . .

pH - remove the sensor wetting cap and slide the vent sleeve to expose the fill hole.

 $\mbox{D.O.}\ -\ \mbox{remove the sensor wetting cap.}$  Move the sensor in a gentle circular motion when measuring.

Cond - immerse probe to halfway point in solution.

Press mode, read, cal or M to turn meter on and start measurement. Place sensor into solution. Automatic endpoint detection freezes the display when plateau is reached; to manually endpoint press read. Press read again to start new measurement.

Continuous measurement may be selected by pressing and holding **read** for 4 seconds. (In this mode battery consumption is higher and the meter will not automatically switch off when not in use.)

Return to auto endpoint by pressing and holding **read** for 4 seconds.

After use, close the filf hole (pH) and replace the wetting cap (pH and DO).

# Calibrating ...

For greater accuracy, calibrate the meter regularly.

#### 1 point

Place the sensor in the calibrating medium:

pН	pH 7 buffer	7.00 pH (at 25°C)
Cond	Hold in free air	0.00 μS
TDS	Hold in free air	0.00 mg/L
DO	Zero oxygen solution	0. %O <sub>2</sub>

(mg/L O<sub>2</sub> is calibrated in %O<sub>2</sub> mode).

Press cal – cal 1 is displayed. After endpointing the display automatically updates to the calibrated value shown, or the temperature compensated value.

If recad is pressed after Cal 1 update, the meter assumes one point calibration only is required. Samples can now be measured.

#### 2 point

Place the sensor in the second calibration medium:
pH pH 4 or 10 buffer 4.00 or 10.01 pH (at 25°C)
Cond Cond std. A or B 1413 µS or 12.88 mS
TDS Cond std. A or B 706 mg/L or 6.44 g/L
DO Hold in air 10 mm
above fresh water 100 %O,

Press *cal* – cal 2 is displayed. After endpointing the display automatically updates to the calibrated value shown or the temperature compensated value.

#### Dissolved oxygen only

In practice, cal 2 is required more frequently than cal 1, this is achieved by pressing *cal* twice to advance directly to cal 2.

In mg/L O₂ mode, correction may be made for salinity and barometric pressure. Press cal - 100 is displayed. Use ▲ and ▼ to adjust the display according to these tables:

ļ.	Cond (mS)	Salinity (g/L)	Setting	
į.	<u>.</u> .	0.0	\$00 T	
	. 6	2.5	97	
	i 11 ·	5.0	95	
	15	7.5	93	
	20	10.0	90	
	24	12.5	88	
,	27	15.0	85	
•	31	17.5	82	
٠.			<u>.</u>	
ı	34	20.0	B1	
	34 Barometric	20.0 Setting	B1 Barometric	Setting
	<u> </u>			Setting
	Barometric Pressure (mm)		Barometric	Setting 95
	Barometric Pressure (mm)	Setting	Barometric Pressure (mm)	-
	Barometric Pressure (mm) 600	Setting 79	Barometric Pressure (mm) 720	95
	Barometric Pressure (mm) 600 620	Setting 79 82	Barometric Pressure (mm) - 720 - 740	95 97
	Barometric Pressure (mm) 600 620 640	79 82 84	Barometric Pressure (mm) 720 740 760	95 97 100
	Barometric Pressure (mm) 600 520 640 660	79 82 84 87	Barometric Pressure (mm) 720 740 760 780	95 97 100 103

e.g. if salinity = 5 g/L setting = 95
if pressure = 740 mm setting = 97
for both parameters, setting will be 95 x 97% = 92.

# Using the Memory ...

5 measurements can be stored in the memory.

Entering a reading into memory

Press M when measurement has endpointed.

M 1 (or M 2 – M 5 if readings have already been stored) is displayed indicating that the reading has been saved. Flashing M indicates memory is full.

#### Recalling memory

Press R – the last saved measurement is displayed. Press R again to recall the previously saved measurement. MR 1 to MR 5 indicates which saved measurement is being displayed.

#### Clearing last memory

In memory recall mode (MR) press M to clear the measurement being displayed. Only the last entered measurement can be cleared, i.e. if 4 measurements are saved, the M 2 cannot be cleared without first clearing M 4 and M 3.

#### Clearing all memory

Press Runtil M C is displayed, then press M. All memory will be cleared.

# Changing Mode . . .

Press **mode** to enter the second function of the sensor, i.e. pH to mV, conductivity to TDS, %O<sub>2</sub> to mg/L O<sub>3</sub>.

Press mode again to turn the meter off.

# Display Codes/Problem Solving . . . Using the Test Plug

The test plug is provided to test the meter.

Replace the sensor with the test plug and press read. The display should read:

7.00 pH ± 0.5, 25°C ± 2

F1 - measurement out of range: check that sensor in tip is immersed in solution and the wetting cap is removed (if applicable)

E2 - cal 1 out of range ) check correct calibration

E3 - cal 2 out of range ) medium is used.

Condition/renew sensor

E4 - sensor disconnected

low battery voltage

low electrode slope: condition/renew sensor (pH only)

Default calibration values in memory. Sensor not calibrated (2 point), or battery replaced since last calibration

O manual endpoint selected (continuous measurement)

auto endpoint selected

keypad not responding - replace battery and recalibrate.

#### Operating Hints . . .

Use distilled water when transferring from one solution to another.

 Response time is a function of the sensor and the solution. If the solutions are at different temperatures (or ionic strength – pH only) allow more time for the sensor to respond.

3. Avoid handling the sensor tip.

Make sure no large air bubbles are trapped under the sensor when making measurements.

 Do not use calibration standards after the expiration date.

 Wetting caps should contain: pH – pH 7 buffer; DO – distilled water.

For greatest accuracy calibrants and samples should be at the same temperature.

 pH – keep the electrode filled with the appropriate fill solution to prevent reading drift.

 Conductivity – the sensor shield and probe should be kept clean. Make sure no air bubbles are in the cell chamber during measurement.

10. Dissolved oxygen – the sensor can be removed for several hours as a rechargeable battery in the sensor will maintain polarization. For longer periods the sensor should remain connected to the meter to maintain polarization and recharge the sensor battery.

# Reordering Information ...

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# Meter Specification . . .

Temperat	ure compensation:	0 ~ 100°C
	pH Conductivity/TDS	0 - 50°C
:	Oxygen	0 - 40°C
	Range	Resolution
Temp	~0.5°C - 100°C	0.1°C
pH	0 - 14 pH	0.01 pH
l mV	0 - ± 1000 mV	1 mV
Cond	0.00 - 19.99 μS	0.01 µS
COIN	20.00 μS - 199.9 μS	0.1 μS
	200 - 1999 µS	tμS
Į.	2.00 - 19.99 mS	0.01 mS
TOS	0.00 - 10.00 mg/L	0.01 mg/L
103	10.0 - 100.0 mg/L	0.1 mg/L
i	100 = 1000 mg/L	1 mg/L
1	1,00 - 10.00 g/L	0.01 g/L
200	0 - 200% O,	1%
DO	0 = 200 /a O <sub>2</sub>	0,1 mg/L

Auto switch off; it not operated for 10 minutes after endpoint.

Memory: up to 5 measurements can be stored.

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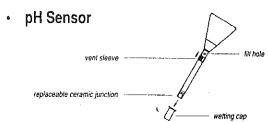
Coming Incorporated Science Products Division Coming, New York 14831 USA Tel: 1-607-737-1667

473655 Rev. A. 3/89

# ensor Information

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and leaded pH, DO and Conductivity Sensors



#### For optimum performance:

- Before use remove wetting cap from tip of sensor, and stide the vent sleeve to expose the fill hole.
- Make sure that the fill solution is not more than 25 mm (1 inch) below the fill hole. Add KCI solution if necessary.
- 3. Gently tap the sensor to remove any air bubbles at the ceramic junction.
- Condition the new sensor by soaking in pH 7 buffer for 2 hours. Prolonged soaking is not recommended.
- Calibrate and measure samples as described in the M90 instructions. Allow sufficient time for the sensor to stabilize when measuring samples of different temperatures, or of low ionic strength. Manual endpointing is advised with these samples.
- After use, check the level of fill solution, reposition the vent sleeve to cover the fill hole, and replace the wetting cap containing pH 7 buffer (if the sensor will not be used again for more than 2 days, we recommend using saturated KCI in the wetting cap).

#### Precautions and Limitations:

- 1. Do not wipe the sensor tip blot dry with a lint-free tissue.
- Do not use KCl saturated with AgCl as this may damage the reference element.
- Do not leave the sensor in organic solvents, strong basic solutions, concentrated fluoride solutions, or hydrofluoric acid for extended periods. Measurements made in these solutions should be taken quickly and the sensor rinsed immediately with distilled water. After rinsing, soak in pH 7 buffer for 2 hours.
- Do not measure solutions that exceed a temperature range of 0 – 100°C.

# Maintenance and Troubleshooting:

Prolonged use and ageing may reduce performance i.e. slow response, low slope values, continuous drift or erratic readings. These may be caused by: Air in junction – remove air bubbles by gentle tapping.

Excess KCI crystals – KCt crystals may build up and settle on the sensor tip, or the KCI may become discolored. Remove the old fill solution and use warm distilled water to dissolve the crystals. Remove water and refill using fresh KCI solution.

Blocked junction – KCl crystals can block the junction. To test for this, blot the tip dry and air dry for one hour. If no KCl crystals appear at the tip of the sensor the junction is blocked. Remove the ceramic junction using tweezers, and insert new junction (Cat. 477269). Tap gently to remove any air bubbles. Contaminated pH bulb – i.e. protein/oil contamination.

Protein - soak the sensor in 10% pepsin solution adjusted to pH 2 with HCl

tor 30 minutes. Hinse with restilled water and soak in pH 7 buffer for 2 hours.

Oil – wash sensor tip with vater acetone solution. *Do not* soak the sensor in acetone solution may cause the seals to deteriorate. Rinse with distilled water and so.

A 7 buffer for 2 hours.

#### Installation:

DO membrane caps are fragile. Handle with care to prevent damage. The sensor is shipped dry and must be filled before use. Unscrew the membrane cap from the sensor. If the silver/gold tip is tarnished clean carefully using electrode cleaning compound or silver polish, paying particular attention to the gold cathode. Rinse tip with DO electrolyte, and fill membrane cap, avoiding air bubbles. Hold the sensor vertically and gently screw the membrane cap onto the sensor, allowing surplus electrolyte to run out. Fit sensor to the meter and allow 1 hour minimum for polarization. Calibrate as described in M90 instructions.

#### For optimum performance:

- Before use remove wetting cap from tip of sensor.
- For immediate use the sensor should be kept connected to the meter.
  The sensor may be removed for up to 3 hours as a rechargeable battery
  in the sensor will maintain polarization. For extended storage remove
  the membrane cap and rinse with water, and clean the sensor tip. Store
  dry with the membrane cap loosely fitted. Do not fit wetting cap.
- When making measurements the sample should be stirred at a constant speed i.e. approximately 20 cm/second (8 inches/second).
- 4. Allow sufficient time for the sensor to stabilize when measuring samples of different temperatures – in some cases this can be several minutes. Manual endpointing is advised with these samples. Make sure the sensor is immersed to a depth of at least 40 mm (1.5 inches) to cover the temperature sensing element.
- After use replace wetting cap containing distilled water to prevent electrolyte from drying out.
- Regular maintenance is important to ensure optimum performance.
   Replacement of membrane caps depends on usage we recommend replacement every 2 to 4 weeks.

#### Maintenance and Troubleshooting:

If the sensor will not calibrate, or becomes sluggish or erratic:

- The silver/gold sensor tip may become tarnished with time. For optimum
  performance clean tip and refill cap every 2 weeks as described in
  Installation.
- The zero oxygen solution will absorb oxygen if left exposed to air and this will cause inaccurate calibration. Use fresh zero oxygen solution.
- Make sure there are no air bubbles inside the membrane cap when filling with DO electrolyte. Check by looking up through the membrane from the bottom of the sensor.
- Check the membrane for damage and replace with new cap (Cat. 473626) as necessary.

Conductivity Senso

clear plastic shield - only remove for cleaning

#### For optimum performance:

- 1. Make sure the clear plastic shield is in place when measurin
- When measuring make sure the solution is above the cell ch and below the vent hole.
- To prevent carryover from high to low conductivity solutions distilled water between measurements.
- Make sure the cell chamber is bubble free when measuring bubbles, immerse probe in the solution at an angle and then vertical position.
- Allow sufficient time for the sensor to stabilize when measuri different temperatures. Manual endpointing is advised with the
- 5. The sensor is not recommended for low ionic strength solution
- 7. Clean the probe and shield with distilled water after use.

# General Troubleshooting for all Sensors

- 1. To verify meter is working check using the test plug.
- If the sensor connector becomes damaged or wet the display E4 when a sensor is connected.
- If the temperature sensing element becomes damaged the tridisplay may read E1 when a sensor is connected.

# Ordering Information:

item

pH sensor
pH electrode fill solution, 3 x 5 mL
pH 7 buffer sachet (pack of 30)
pH 4 buffer sachet (pack of 30)
pH 10 buffer sachet (pack of 30)

pH multipack, pH 4, 7 and 10 (pack of 30 assorted sachets)

Buffer solution pH 4.00, 2 x 500 mL (red) Buffer solution pH 7.00, 2 x 500 mL (yellow)

Buffer solution pH 10.01, 2 x 500 mL (blue)

Buffer rainbow pack, pH 4.00, 7.00 and 10.01 (2 x 500 mL of each Replaceable ceramic junctions (pH), pack of 3

DO sensor

DO electrolyte, 3 x 5 mL Zero oxygen solution, 500 mL DO membrane replacement kit, pack of 2 p0, electrode cleaning compound

Conductivity/TDS sensor

1413 µS conductivity standard, 500 mL 12.88 mS conductivity standard, 500 mL

Rinse solution sachet (pack of 30)

Corning Incorporated Science Products Division Corning, New York 14831 USA Tel: 1-607-737-1667 Technical Information Center: 1-(800)-222-7740

# ATTACHMENT 11J LYSIMETER MONITORING PLAN AND PROCEDURES

#### ATTACHMENT 11J

#### Lysimeter Monitoring Plan and Procedures

#### 11J.1 Lysimeter Monitoring Plan and Field Procedures

One sample from each lysimeter will be collected quarterly for chemical analysis provided a sufficient volume of fluid can be obtained. The two lysimeters are located between the sedimentation basin and the perimeter dike of Cell I, as shown on Figure 11J-1. To maintain consistency with the previously collected background samples, it is recommended that samples are identified as LY-1A-OP and LY-1B-OP. A schematic of the Teflon® lysimeters is shown on Figure 11J-2, and a description of their operation and sampling methods are found on Figure 11J-3. Collected samples will be preserved on ice until delivery to the laboratory for analysis, if refrigeration is required.

All samples will be properly labeled according to EPA Chain-of-Custody requirements using a sample tag as shown on Figure 11J-4, or equivalent. The chain-of-custody tag will be attached to the sample container at the time the sample is collected. The sample container will be sealed tightly. The tag will be filled out in ink and in legible handwriting.

The Chain-of-Custody Form describing the sample will be drawn up by the sampler. This form will accompany all samples, and be signed and dated by the transferee at the time the samples are released. The individual accepting the sample will also sign and date the form acknowledging the receipt of the letter that describes the transfer.

The parameters that have been monitored in the background monitoring program are listed in Table 11J-1. Because the lysimeters are designed to monitor Cell I, new parameters are not being added to this monitoring plan. Sample container types, preservation methods, holding times, detection limits, and analytical methods for the operational monitoring are listed in Table 11J-2.

A detailed laboratory QA/QC program is provided in Attachment 11A.

#### 11J.2 Statistical Evaluation of Lysimeter Monitoring Data

As soon as possible following each quarterly sampling event, a statistical evaluation of a portion of the data will be conducted to determine if a significant increase above background concentrations has occurred. Statistics will be performed on all the parameters listed in Table 11J-1.

The lysimeter data will be evaluated on an intra-point basis, so that operational monitoring data from each lysimeter will be compared to the background data set from that same sampling location. Follow the procedures outlined below for statistical evaluation. If the reported concentration results in an exceedance of that test statistic, then the procedure outlined in Subsection 11,10 will be followed.

- Tabulate, evaluate, and reduce the existing background data. Review data to determine completeness and to determine if sample locations were consistent and clearly defined. Review any additional background data in the same manner when background data collection is complete.
- 2. Revise, if necessary, the estimated quantitation limits (RDLs) for each constituent. RDLs reported by analytical laboratories may change with time. Use the largest detection limit for each parameter wherever a correction for censored data is needed. Using the largest reported RDL value for each constituent is appropriate because the maximum value sets the level of accuracy that can be attained in future monitoring, even if RDL values decrease in the future.
- 3. If the background data set is 100 percent censored, do not perform statistics. The actual level of detection will serve as the trigger value for resampling. In this case, an operational monitoring sample result which exceeds the analytical detection limit will be confirmed by collecting an individual sample at that location and analyzing for the parameter which exceeded background. This measure is being taken in order to rule out laboratory error as a source of the detection. If the analyte is not detected in the confirmatory sample, no further action will be taken. If the analyte is detected, resample at that location in quadruplicate. If any of the reported concentration exceed the RDL, then follow the procedures listed in Subsection 11.10.3.
- 4. Assess the underlying statistical distribution of the data, and correct for log normality if necessary. After the first round of statistical evaluation has been completed, this step will consist of transforming the current data, if necessary, based on the previous evaluation. As recommended in the February, 1993 USEPA Guidance document, assess normality by constructing probability plots.

Follow the attached guidance for constructing probability plots. The plotted points will approximate a straight line if the data are normal. Construct probability plots of the log-transformed and the raw data for each parameter. Compare the plots and decide which representation of the data is closer to the

normal distribution. If the log transform of the data is selected as an appropriate transformation, transform all background and operational data for that sample point for that parameter prior to conducting any statistical tests on the data. State whether the statistical test was conducted on raw or transformed data in all reports regarding the data.

- 5. Inspect the data set for outliers. Conduct formal testing for outliers only if a reported concentration is orders of magnitude higher than the rest of the data set. Follow the procedure for outlier testing presented at the end of this attachment. The outlier test assumes that the data other than the outlier follows a normal distribution. Therefore, if the data set is log-normally distributed (see number 3 above), conduct the outlier test on the log-transformed data. Correct or remove an outlier from the data set only if the value can be identified as:
  - a) an error in transcription or dilution;
  - b) a documented error in an analytical procedure or report of matrix interferences in the procedure; or
  - c) some other factor from those listed in the RCRA guidance (USEPA, 1989; USEPA, 1993).

In the event an outlier can be verified, obtain MDNR permission before removing the outlier from the data set. If no obvious cause can be identified for a value being an outlier, it will remain in the operational data set used for statistical evaluation unless MDNR approval is obtained to remove it.

- 6. Inspect the current round of data for nondetects. If a parameter was reported to be below the RDL for that round, then do not perform a statistical test with that data (i.e., do not perform a statistical evaluation to determine if a nondetect represents an exceedance of background). Add the analytical result to the database for that sample point. This approach is being taken because it is reasonable to assume that a nondetect cannot represent an exceedance of background.
- 7. Evaluate the degree of censorship in the data, and select the appropriate statistical test based on this evaluation. After the first round of statistical evaluation has been completed, this step will consist of evaluating whether the statistical test used during previous rounds remains an appropriate choice for the data set. The following steps will be followed in determining how censored data will be handled and in choosing the statistical test to be performed for each sampling point:
  - a) If the percentage of nondetects in the database for the sample point is less than 15 percent, substitute of a value of 1/2 the RDL for all nondetects and calculate a prediction interval according to the procedure included at the end of this Attachment.
  - b) If the percentage of nondetects in the database for the sample point is between 15 and 50 percent, use Cohen's or Aichison's adjustment to calculate the mean and standard deviation of the background data.

Use these adjusted statistics to calculate a prediction interval. Follow the procedures presented at the end of this attachment to determine which of Cohen's or Aichison's adjustment should be used. Calculate the prediction interval according to the presented procedure.

- c) If the percentage of nondetects in the database for the sample point is between 50 and 90 percent, use the Wilcoxan Rank-Sum Test to compare operational monitoring results to background data. Procedures are attached.
- d) If the percentage of nondetects in the database for the sample point is 90 percent or greater, calculate a Poisson prediction limit. Procedures are included at the end of this Attachment.
- e) If the percentage of nondetects in the background data set is 100 percent follow the procedure listed above in step number 3.
- 8. If the statistical test performed for step number 7 above indicates that an exceedance has occurred, follow the steps outlined in Subsection 11.10 of the Permit.

TABLE 11J-1			
CELL I LYSIMETER MONITORING PARAMETERS			
	Scan 8 Phenolics		
2-Chlorophenol 2-Methyl-4,6-dinitrophenol 2-Nitrophenol 2,4-Dichlorophenol 2,4-Dimethylphenol 2,4-Dimitrophenol 2,4-Dinitrophenol 2,4-Dinitrophenol			
S	can 7 Polynuclear Aromatic Hydro	arbons	
Acenaphthene Acenaphthylene Anthracene Benzo(A)anthracene Benzo(A)pyrene Benzo(B)fluoroanthene	Benzo(G,H,I)perylene Benzo(K)fluoranthene Chrysene Dibenzo(A,H)anthracene Fluoranthene Fluorene	Indeno(1,2,3-CD)pyrene Naphthalene Phenanthrene Pyrene	

TABLE 11J-2
LYSIMETER SAMPLING PARAMETERS AND METHODS

Parameter	Analytical Method <sup>1</sup>	Estimated Detection Limit	Container & Preservative	Holding Time
2,4,5-Trichlorophenol	8270	10 µg/L	AG, R	E
2,6,4-Trichlorophenol	8270	10 µg/L	AG, R	E
2,4-Dichlorophenol	8270	10 μg/L	AG, R	E
2,4-Dimethylphenol	8270	10 µg/L	AG, R	E
2,4-Dinitrophenol	8270	50 µg/L	AG, A	E
2-Chlorophenol	8270	10-µg/L	AG, R	E
2-Nitrophenoi	8270	10 μg/L	AG, R	Е
4-Nitrophenol	8270	50 μg/L	AG, R	E
2-Methyl-4,6-dinitrophenol	8270	50 µg/L	AG, R	E
Pentachlorophenol	8270	50 µg/L	AG, R	E
4-Chloro-3-methylphenol	8270	10 μg/L	AG, R	E
Phenoi	8270	10 μg/L	AG, R	E
Acenaphthene	8310	1.0 μg/L	AG, R	E
Acenaphthylene	8310	1.0 μg/L	AG, R	E
Anthracene	8310	1,0 µg/L	AG, R	E
Benzo(A) anthracene	8310	2.0 μg/L	AG, FI	E
Benzo(A) pyrene	8310	2.0 μg/L	AG, R	Ε
Benzo(B)fluoranthene	8310	2.0 μg/L	AG, R	E
Benzo(G,H,I)fluoranthene	8310	5.0 μg/L	AG, R	E
Benzo(K)fluoranthene	8310	2.0 μg/L	AG, R	
Chrysene	8310	1.0 μg/L	AG, R	E
Dibenzo(A,H)anthracene	8310	5.0 μg/L	AG, A	·E
Fluoranthene	8310	1.0 μg/L	AG, R	E
Fluorene	8310	1.0 μg/L	AG, R	ε
Indeno(1,2,3-CD)pyrene	8310	5.0 μg/L	AG, R	E

# **TABLE 11J-2**

# LYSIMETER SAMPLING PARAMETERS AND METHODS

Parameter	Analytical Method	Estimated Detection Limit	Container & Preservative	Holding Time
Naphthalene	8310	1.0 µg/L	AG, R	E
Phenanthrene	8310	1.0 μg/L	AG, R	Ε
Pyrene	8310	1.0 μg/L	AG, R	E

#### NOTES:

"Test Methods for Evaluating Solid Waste," SW-846, Third Edition.

2 USEPA Methods 600/4-79/200.

P Plastic.

G Glass.

R

Refrigeration.
Nitric acid to pH <2. Ν

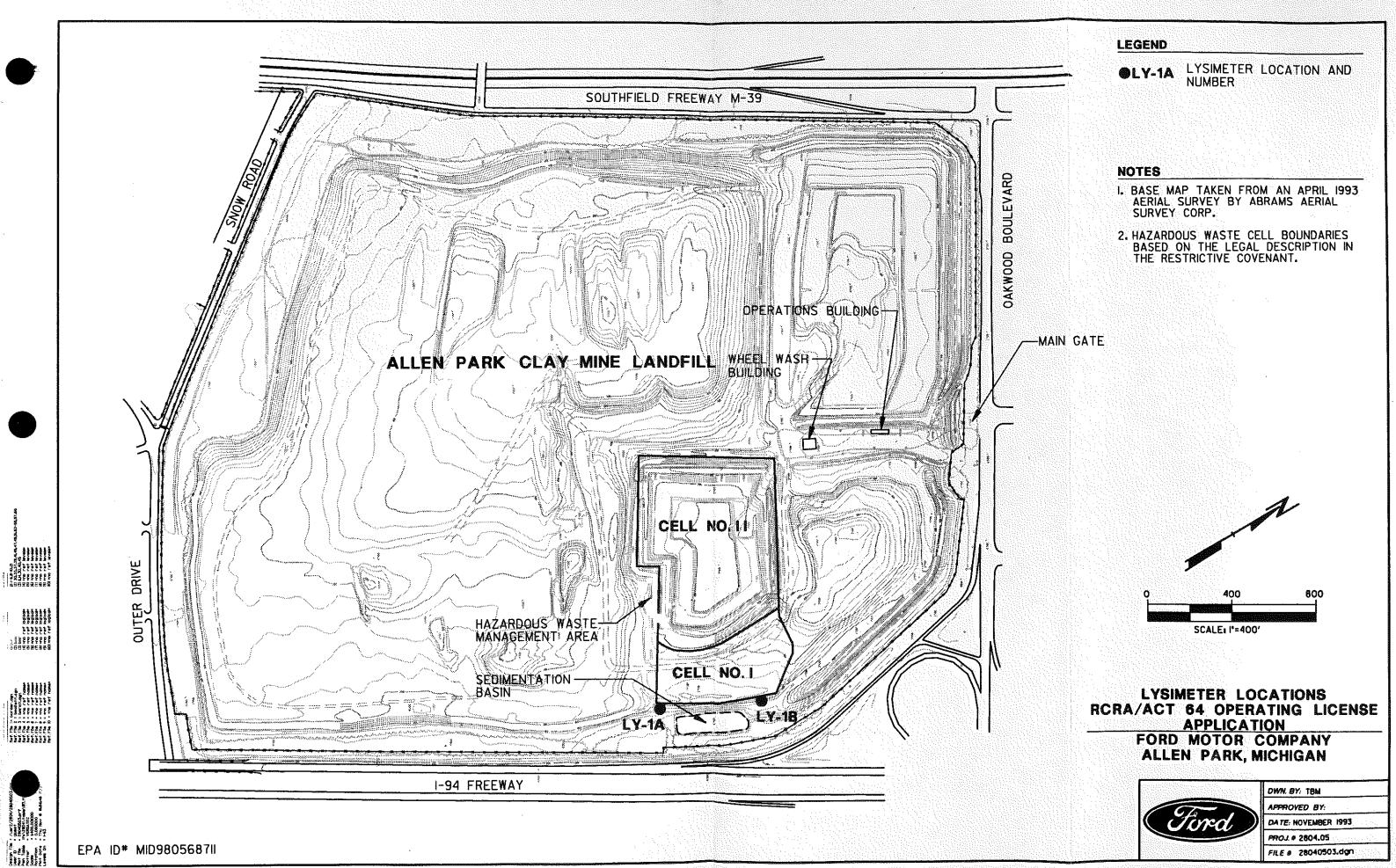
Т Teflon&lined cap.

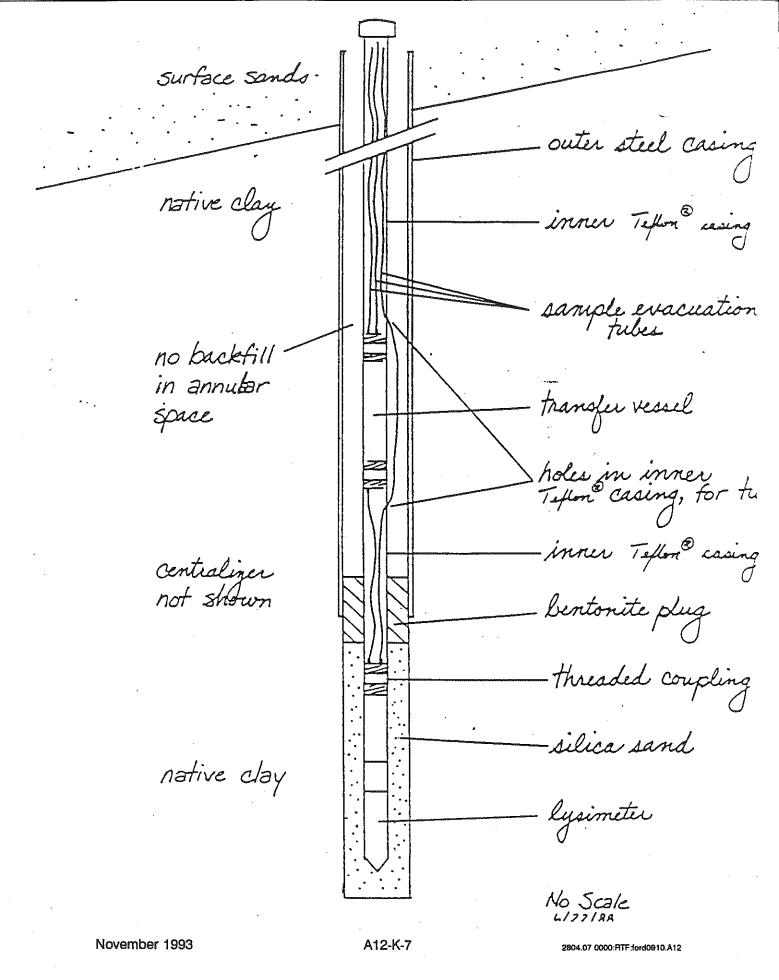
7 days to extraction and 40 days from extraction. Sulfuric acid to pH <2. Ε

S

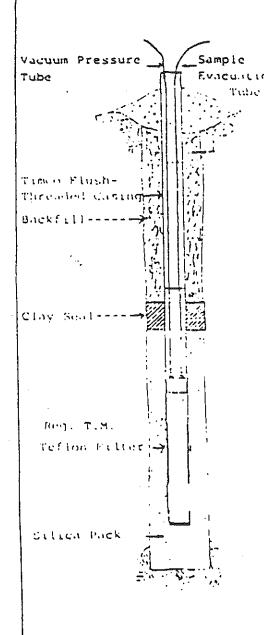
AG Amber glass.

Hydrochloric acid to pH <2. С





#### INSTALLATION OF A TIMO LYSIMETER



Reg. T.M. "Teflon" Dupont Registered Trademark

- 1. Check to make sure all joints and fittings on the lysimeter and tubes are tight. This can be casily ascertained by immersing the entire unit in distilled water and applying no more than 20 lbs. of air pressure. Observe all connections Evacuation and joints for air bubbles except for the purous filter portion. The porous filter portion should give off bubbles over the entire surface. If any leaks are observed at any point other than the porous filer, the connections should be slightly rightened and checked again. If leaks are still observed, Teflon tape should be used on the threads and the unit re-assembled and checked. Also check for leaks at the plug, stop-cock, clamp, vacuumpressure gauge or other devices to be used at the top of the tubes at the top of the borehole. 2. For a Two inch diameter lysimeter, drill a six unch borehole or larger. It is recommended that as a minimum there be at least one and onehalf inches on each side of the lysimeter for the silica pack. (If difficulties are encountered in maintaining an open borehole, use casing to hold back the material. After installation of the silica pack and lysimeter, pull back the casing and install a bentonite plug.)
  - J. Make a slurry of silica using 150 ML. distilled water and 450 grams of 200 mesh silica flour: suggested use, 50-60 lbs. of silica flour per lysimeter. (2 gals. water to 50-60 lbs. silica flour). Care should be taken to blend water and silica completely, make certain that no "lumps" exist.
  - 4. Pour part of the silica slurry into the borchole just prior to installation of the lysimeter. (Minimum of 1) inches in the bottom for tube type, 4 inches for cup type).
  - 5. Place the lysimeter into the borehole, care being taken to ensure that the lysimeter is centrally located. A minimum of 1 inches of silical pack around the annulus area of the unit is essential. We suggest that the lysimeter should be ordered with a threaded top plug which allows use of TIMCO flush threaded riser pipe with attached centralizate to ensure centering.

TIMCO MFG.

- 6. Pour the balance of the silica slurry eround the lymmeter ensuring that the entire unit is completely covered. This will allow for any "settling" or slumping of the silica pack an moisture is withdrawn.
- 7. Place a bentonite seal above the silica pack followed by tamped backfull. A bentonite surface seal or other type of seal is also recommended. The best of course, is a steel security cover with a lock, set in concrete.
- 8. Clamp off or plug the sample evacuation tube. (If Terlor tubes are being used, one a Teflor plug of a Teflor stop-cock of crimp no will do damage to the tube.)
- 9. Using a vacuum-pressure hand pump or a portable vacuum pump, apply 18 to 21 inches of mercury. We recommend that a stop-cock be installed in the vacuum-pressure line at the surface as well as a wacuum-pressure gauges. If a gauge is not installed, there is no accurate way of determining the amount of initial vacuum or how much vacuum may be left at a later time. A stop-cock ensures no loss of vacuum as the pump is being disconnected, especially with the Teflor tubing, as previously mentioned may not be crimped or clamped off without damaging the tube.
- 10.7 When the vacuum-pressure gauge indicates less than 10 inches of mercury (approximately 24 hours), a sample should be attempted.
- 11. SAMPLE FETRIEVAL OF THE SHALLOW TYPE: Using a sample flask that is more than adequate for the volume of the lysumeter, with a two fole stopper it the top, insert and occurs the sample or evacuation that in one of the holes, making certain that the tube clears the bottom of the stopper by at least i of an inch. Using another tube of adequate length, altach one end to the vacuum pump and insert the other end of the tube thru the other hole in the stopper (have tube flush with the bottom of the stopper) and apply vacuum. Vacuum should be repeated until no further sample is obtained. Remove the tubes from the sample flask, repeat procedure number 0 thru 10. Discard from the first sample OR CAMPLES, approximately 10% of the volume of the water used to mix the silica pack. Depending upon soil moisture and other factors, the second and subsequent samples may take up to 48 hours or longer to obtain a full sample. A vacuum pressure of less than 10 inches will indicate that a sample should be taken. We suggest that the installition be checked each 26 hours.
- 12. SAMPLE RETRIEVAL UTILIZING THE THANSFER VESSEL AT DEPTHS BELOW TO FEET. Pefore installation of the lysimeter attach the transfer vessel 5'-10' above the lysimeter as in figure No. 2. Follow procedures 1 thru 10. Using a sample flask that is more then adequate for the volume of the lysimeter, insert and secure the sample or evacuation tube in a vented sample flask. Attach the gas or pressure tark to the vacuum-pressure tube attached to the lysimeter setting the pressure not to exceed 21-5 lbs., forcing the sample into the

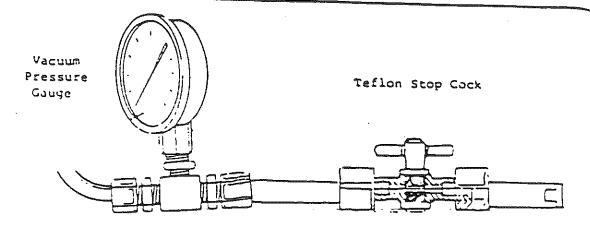
transfer vessel. Py using this amount of lbs. of pressure the lysimeter will not be overpressurized causing the silica pack to separate from the teflon filter. Then attach the gas or pressure tank to the second pressure tube which is attached to the transfer vessel. Set the pressure at about .42 lbs. per foct of depth. Apply pressure until all sample is obtained. Discard from the first sample OR SAMPLES, approximately 20% of the volume of the water used to mix the silica pack. Prpending upon soil moisture and other factors, the second and subsequent samples may take up to 48 hours or longer to obtain a full nample. A vacuum pressure of less than 10 inches will indicated that a sample should be taken. We auggest that the installation be checked each 14 hours.

NOTE: We further recommend that a 99.5% pure value, be used in the salice " pack. Chemical unalysis of one-half of one per cent impurities are available upon request. For critical installations, pure silica is avaiabable but is extremely expensive.

FURTHER MOTE: It is vacuum is used in a tweimeter restallation loss then 26 feet in depth, or where a power source is available with controls to maintain a constant vacuum (a sample flack of sufficient volume should be used on the vacuum-pressure line to prevent damede to the pump or controls. The installation should be choosed each of hours).

#111282 Timco

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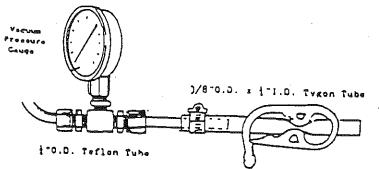
End Plug

For lysimeter installations, the Vacuum-Pressure gauge is adapted to a teflon T fitting which is attached to the teflon i" 0.0. tube. The teflon tube is but and grooved with a groover to allow the fittings to tighten properly so the tube does not slip out. At this point a teflon stop-cock is installed, then a teflon plug is inserted in the end of the tube.

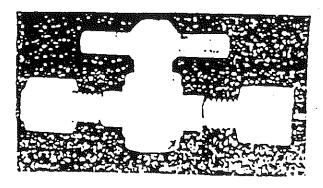
All fittings should be tightened with a wrench.

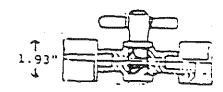
Pressure source tubes and sample evacuation tube should each have a teflon stop-cock and end plug installed.

while it is desireable to ittuch a teflon stopcock it the end of the tube as mentioned above, shown below as a cost effective option. A piece of 3/8" O.D. x i" I.D. tygon tube is placed over the teflon tube with a steel base clamp holding it in place. The end of the tygon tube is sealed off with a plastic gripping clamp.



# TEFLOW STOPCOCK



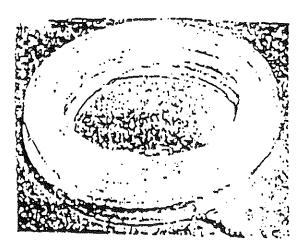


frection molded of chemicallay tract Tellon-Ita, the stopcock has a one piece hundle and stem construction.

The atopolick has a quarter tarn open-close greation with a straight through writice.

There are no menal or other plastic parts which could corroda in caustic environments.

TEFLOR THE TUBING



Available in  $1^{+}$  O.D. x  $1/8^{+}$  I.D. in continuous lengths. It is chemically inert, even with the most explic solvents. Out and pre-grooved for use with tellon fittings.

# SAMPLING PROCEDURES FOR THE TIMOO TUBE LYSIMETER WITH TRANSFER VESSEL

- 1. Using the stop-cocks shut off the pressure tube to the transfer vessel and the sample tube to the transfer vessel.
- 2. Attach the hand pump to the lysimeter pressure tube, which has the pressure gauge attached. Make sure pump is attached at the vacuum end of the pump. (See attached diagram)
- 3. Making sure the stop-cock on the lysimeter pressure tube is open apply 18 to 21 inches of mercury.
- 4. Reading the gauge, once it reaches 18 to 21 inches of Hg. shut off the stop-cock on the lysimeter pressure tube.
- 5. All three lines should be shut off now and a vacuum of 19 to 21 inches of Hg. should read steadily on the gauge. In approximately 24 hours, if the pressure gauge reads less than 10 inches of Hg. a sample should be taken.
  - 6. <u>Sample Retrieval Using Transfer Vessel</u>
    Open all stop-cocks. Insert the transfer vessel sample tube into a vented sample flask or sampling container.
  - 7. Attach the pressure end (see diagram) of the hand pump to the lysimeter pressure tube. Apply between 2½-5 psi of pressure forcing the sample into the transfer vessel. Keep this low pressure applied until you are sure the complete sample has been transferred. Do not use higher pressure since damage to the silica pack surrounding the lysimeter could occur.
  - 3. Now attach the hand pump at the pressure end to the transfer vessel pressure tube. Apply pressure at a rate of .42 lbs. per foot of depth. (Example: 50 ft of vertical depth equals 21 lbs.)
  - 9. Apply pressure until all sample is obtained. Discard the first sample or samples until 30% of the volume of distilled water used in mixing the silica pack is accounted for. Example using 2 gallons (7571 ml) to mix the silica slurry would mean that the first .6 gallons (2271 ml) of sample retrieved should be discarded since it contains simply distilled water from the silica pack.

10. Start at step one. Depending on soil moisture and soil type the second and subsequent samples may take 24 hours or longer to obtain a full sample. When the vacuum gauge drops below 10 inches of Hg. the sample should be take. It is suggested that the installation be checked once every 24 hours once a vacuum is applied until a full sample is obtained.

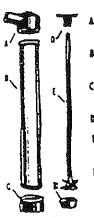
# PAR " UTILITY HAND PUMPS

#### General Information

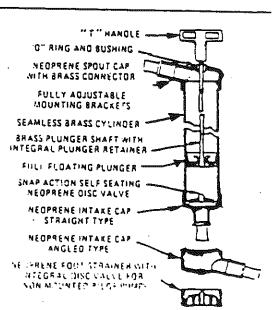
PAR rumps never need coaxing or priming, full suction on the first stroke. Brass cylinders, high-quality oil resistant plungers, intake and discharge caps for the long life. Pumps can be used in any position. Available in 1-1/4" diameter cylinders.

# Specifications

	STPOKES				•	
MODEL	PER GAL.	CYL. DIA.	STROKE LENGTH	OVERALL LENGTH	HOSE CONNECTION	YEIGHT
33799-0000	. 26	1-1/4"	7-1/2"	12"	1/2"	2.0



- A.One-piece neoprene cap with up-angled spoul.
- B.Heavy seamless brass cylinder; rolled ends.
- C.Neoprene foot strainer with integral valve.
- B\_Molded handle
- f. 3 rass shaft with integral plunger retainer.
- F. Full-floating, frictionfree neoprene plunger, the most efficient plunger in any sump.



#### Maintenance

To disassemble pump for cleaning and repair, pull handle out approximately 4" and place screwdriver carefully under bottom of spout cap and over outside of cylinder roll. Lift up on screwdriver and pry off spout cap. Pull out plunger shaft

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assembly. Lift full floating plunger off seat and clean between cup and seat. Remove intake cap in same way as spout cap. Take disc valve from cylinder and clean under neoprene flapper. Run rag through cylinder for cleaning.

To reassemble pump place disc valve in cylinder, snap intake cap back onto cylinder being sure undercut in cap is over cylinder roll. Insert plunger shaft assembly into cylinder and snap spout cap over cylinder roll.

Oil pump internally and spray with corrosion inhibitor or light oil for storage.

TIMEO MEG.

		Ford Allen Park Clay Mine Ford Motor Company Lysimeter Sample
	Date	
	Time	
	Lysimeter #	
((<))	Analyze for	
	Preserved with	
$\lambda$ //		
// //		
// // \	Samplers Signatures	
// //		Date

EACH SAMPLE TRANSFERRED SHOULD HAVE A LABEL ATTACHED.

SAMPLE LABEL
FORD MOTOR COMPANY
ALLEN PARK, MICHIGAN



DWN. BY: TBM

APPROVED BY:

DATE: NOVEMBER 1993

PROJ. # 2804.05